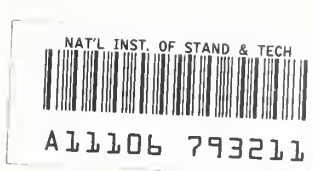
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CALIBRATION LABORATORIES

TECHNICAL GUIDE FOR MECHANICAL MEASUREMENTS

NIST Handbook 150-2G

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NIST HANDBOOK 150-2G

National Voluntary Laboratory Accreditation Program

Calibration Laboratories

Technical Guide for Mechanical Measurements

C. Douglas Faison and
Carroll S. Brickenkamp, Editors

March 2004

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Preface

The Calibration Laboratories Accreditation Program was developed by the National Voluntary Laboratory Accreditation Program (NVLAP) at the National Institute of Standards and Technology (NIST) as a result of interest from private industry and at the request of the National Conference of Standards Laboratories (now the NCSL International). The program's goal is to provide a means by which calibration laboratories can be assessed for competency. This voluntary program is not designed to serve as a means of imposing specific calibration procedures or minimum uncertainties on applicant laboratories; instead, the program allows for all scientifically valid calibration schemes and requires that laboratories derive and document their measurement uncertainties.

To accomplish this goal, NVLAP employs technical experts on a contract basis. They serve as assessors in each of the following eight fields of physical metrology calibration:

- electromagnetic dc/low frequency,
- electromagnetic rf/microwave frequency,
- time and frequency,
- ionizing radiation,
- optical radiation,
- dimensional,
- mechanical, and
- thermodynamics.

NIST Handbooks 150-2A through 150-2H are technical guides for the accreditation of calibration laboratories. Each handbook corresponds to one of the eight fields of physical metrology calibration. They are intended for information and use by:

- NVLAP technical experts in assessing laboratories,
- staff of accredited laboratories,
- those laboratories seeking accreditation,
- other laboratory accreditation systems,
- users of laboratory services, and
- others needing information on the requirements and guidelines for accreditation under the NVLAP Calibration Laboratories Accreditation Program.

NOTE The Calibration Laboratories Accreditation Program has been expanded to cover chemical calibration for the providers of proficiency testing and certifiers of spectrophotometric NIST-Traceable Reference Materials (NTRMs). (See NIST Handbooks 150-19 and 150-21.) Other NVLAP handbooks in the chemical calibration area are expected in the future.

The assessor uses NIST Handbook 150, *NVLAP Procedures and General Requirements*, and the appropriate guides (NIST Handbooks 150-2A through 150-2H) to validate that a laboratory is capable of performing calibrations within the laboratory's stated uncertainties. These technical guides and other relevant technical information support assessors in their assessments of laboratories. Along with inspecting the facilities, documentation, equipment, and personnel, the assessor can (1) witness a calibration, (2) have an item recalibrated, and/or (3) examine the results of measurement assurance programs and round-robins to collect objective evidence.

NIST Handbooks 150-2A through 150-2H supplement NIST Handbook 150, which contains Title 15 of the U.S. Code of Federal Regulations (CFR) Part 285 plus all general NVLAP procedures, criteria, and policies.

The criteria in NIST Handbook 150 originally encompassed the requirements of ISO/IEC Guide 25:1990 and the relevant requirements of ISO 9002 (ANSI/ASQC Q92-1987). These handbook criteria have been updated to incorporate the requirements of ISO/IEC 17025:1999. The entire series of Handbooks 150-2A through 150-2H contains information specific to the Calibration Laboratories Program and neither adds to nor detracts from requirements contained in NIST Handbook 150.

Any questions or comments on this handbook should be submitted to the National Voluntary Laboratory Accreditation Program, National Institute of Standards and Technology, 100 Bureau Drive, Stop 2140, Gaithersburg, MD 20899-2140; phone (301) 975-4016; fax (301) 926-2884; e-mail NVLAP@nist.gov.

Acknowledgments

NIST Handbook 150-2 was first available as a draft covering all eight fields of physical metrology calibration in one volume. It has been separated into eight handbooks to allow easier updating and electronic downloading from the NVLAP web site. The preparation of these documents has been a joint effort, with input from representatives of other government agencies, laboratories, and the private sector. While acknowledgment of their efforts is in order, listing of individual names is impractical. The submissions by individuals and companies offering suggestions for improvement to this document were also very welcome, as were the contributions of those who attended the public workshops.

We thank all the NIST measurement divisions for their work in writing or contributing to the individual handbooks. Listed below are those from the NIST measurement divisions who deserve special thanks for input to Handbook 150-2G, *Technical Guide for Mechanical Measurements*:

- Mr. G. P. Baumgarten (Flow Measurements)
- Dr. Donald G. Eitzen (Acoustics and Vibration)
- Mr. David J. Evans (Vibration)
- Mr. J. M. Hall (Air Speed Measurements)
- Mr. John F. Houser, Dr. Zeina Jabbour, and Ms. Georgia L. Harris (Mass, Volume and Hydrometers)
- Dr. George E. Mattingly (Flow Measurements, Air Speed Measurements, Volume, and Hydrometers)
- Dr. Victor Nedzelnitsky (Acoustics)
- Ms. Jennifer L. Scott (Flow Measurements of Inert Cryogens)
- Mr. Sam Low, (Hardness)
- Dr. Simone L. Yaniv (Force).

Additional thanks go to those who actively participated in the Technical Guide Workshop held November 1993 and to those who served as points of contact within fields of calibration. They include: Ms. Georgia L. Harris, Mr. Norman B. Belecki, Dr. Theodore D. Doiron, Mr. Robert M. Judish, Mr. Thomas C. Larason, Ms. Sally S. Bruce, and Dr. Donald B. Sullivan. A special thanks is owed to Mr. James L. Cigler for work in developing the content and format of this guide, and to Ms. Vanda White for her editorial expertise in making this a readable document.

Above all, we wish to thank Mr. Jon M. Crickenberger, the editor of the first three drafts of this document, for literally hundreds of hours of work in creating this guide. He tasked the contributors to produce the technical content, assembled the results of their efforts into a consistent format, and provided the general commentary. Without Jon's dedicated effort to this monumental task, this guide would never have been published.

NVLAP has edited the individual handbooks and made changes resulting from comments by individuals to earlier draft versions. This editing has been to a different extent for each parameter. Every effort was made to include all pertinent information relevant to an ISO/IEC 17025-derived technical guide.

C. Douglas Faison and Carroll S. Brickenkamp, Editors
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100 Bureau Drive, Stop 2140
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Summary

This guide contains the general technical requirements (i.e., on-site assessment and proficiency testing) of the laboratory accreditation program for calibration laboratories along with specific technical criteria and guidance applicable to mechanical measurements. These technical guidelines indicate how the NVLAP criteria may be applied.

Any calibration laboratory (including commercial, manufacturer, university, or federal, state, or local government laboratory) engaged in calibration in mechanical measurements covered by this handbook may apply for NVLAP accreditation. Accreditation will be granted to a laboratory that complies with the criteria for accreditation as defined in NIST Handbook 150. Accreditation does not guarantee laboratory performance – it is a finding of laboratory competence.

Fields of calibration covered: Specific calibration parameters and related stimulus and measurement devices in areas of mechanical measurement.

Scope of accreditation:

- Calibration parameter(s), range, and measurement uncertainty level
- Types of measuring and test equipment
- Quality assurance system for measuring and test equipment

Period of accreditation: One year, renewable annually.

On-site assessment: Visit by an assessor(s) to determine compliance with the NVLAP criteria before initial accreditation, in the first renewal year, and every two years thereafter. Preassessment and monitoring visits are conducted as required. All calibration parameters or general areas of calibration within the specific scope of accreditation requested will be assessed.

Assessors: Technical experts with experience in the appropriate areas of calibration and quality systems assessment.

Proficiency testing (measurement assurance): Each laboratory is required to demonstrate its capability to successfully perform calibrations as part of on-site assessment or by documented successful completion of an approved Measurement Assurance Program (MAP) or round-robin intercomparison. Proficiency testing may be required for initial accreditation or where other evidence of measurement assurance is not evident. Proficiency testing may also be conducted annually thereafter. Advance notice and instructions are given before proficiency testing is scheduled.

Fees: Payments are required as listed on the NVLAP fee schedule, including the initial application fee, administrative/technical support fee, on-site assessment fee, and proficiency testing fee.

1 General information

1.1 Purpose

The purpose of this handbook is to amplify the general requirements for accreditation by NVLAP of calibration laboratories in the area of mechanical measurements covered by the Calibration Laboratories Program. It complements and supplements the NVLAP programmatic procedures and general requirements found in NIST Handbook 150, *NVLAP Procedures and General Requirements*. The interpretive comments and additional guidelines contained in this handbook make the general NVLAP criteria specifically applicable to the Calibration Laboratories Program.

This handbook does not contain the general requirements for accreditation, which are listed in NIST Handbook 150. Rather, this handbook provides guidelines for good calibration laboratory practices, which may be useful in achieving accreditation.

1.2 Organization of handbook

The handbook is organized in two sections. The first section provides additional explanations of the general procedures and requirements contained in NIST Handbook 150. The second section provides details and guidance specifically for mechanical calibration laboratories.

1.3 Description of Calibration Laboratories Accreditation Program

On May 18, 1992, as a result of the petition and public notice process, the Director of the National Institute of Standards and Technology published in the *Federal Register* a notice of intent to develop the Calibration Laboratories Accreditation Program under the procedures of the National Voluntary Laboratory Accreditation Program . On June 2, 1994, the procedures and general requirements under which NVLAP operates, Title 15, Part 285 of the U.S. Code of Federal Regulations (CFR), were revised to:

a) expand the procedures beyond testing laboratories to include accreditation of calibration laboratories;

b) update the procedures to ensure compatibility with generally accepted conformity assurance and conformity assessment concepts;

c) incorporate international changes, especially with relevant International Organization for Standardization/International Electrotechnical Commission (ISO/IEC) documents (e.g., ISO/IEC Guides 25 (now ISO/IEC 17025:1999), 38, 43, and 58, and the ISO 9000 series); and

d) facilitate and promote acceptance of the calibration and test results between countries to avoid barriers to trade.

Calibration laboratory accreditation is offered in eight fields of physical metrology calibration covering a wide variety of parameters, It includes accreditation in multifunction measuring and test equipment calibrations. Specific requirements and criteria have been established for determining laboratory qualifications for accreditation following prescribed NVLAP procedures. The criteria address quality systems, staff, facilities and equipment, test and calibration methods and procedures, manuals, records, and calibration/certification reports.

On September 18, 1992, a public workshop held at NIST Gaithersburg was attended by a mix of private sector and government personnel. The workshop reviewed a draft handbook, which included general requirements, as well as very specific technical requirements for dc voltage calibrations at all levels. As a result of the workshop, the draft handbook was revised to take the form of a Calibration Laboratories Program Handbook. The handbook included the general requirements for laboratories (using ISO/IEC Guide 25 as a basis), and eight companion Technical Guides covering the specific requirements for each field of calibration offered for accreditation.

On May 18, 1993, a public workshop on the revised draft program handbook held at NIST Boulder was attended by more than 60 industry and government personnel. Comments from this workshop, as well as responses to a survey/checklist mailing, were used to prepare the final draft of the handbook, now entitled *NVLAP Procedures and General Requirements* (NIST Handbook 150), published in March 1994. [A revised NIST Handbook 150 incorporating ISO/IEC 17025:1999 is dated 2001.]

A public workshop for the Calibration Laboratories Technical Guides was held at NIST Gaithersburg, on November 22 through 24, 1993. More than 60 industry and government personnel attended and provided comments on the draft version of the Technical Guide for each of eight fields of calibration. As a result, the eight Technical Guides were incorporated into a draft Handbook 150-2, *Calibration Laboratories Technical Guide*, covering the fields being offered for accreditation. [In 2000, Handbook 150-2 (draft) was divided into eight handbooks, one for each calibration area.]

The need for technical experts to serve as assessors was advertised. The first group of assessors was selected and trained during a four-day session held from November 16 through 19, 1993, in Gaithersburg, using materials developed by NVLAP.

The Calibration Laboratories Accreditation Program officially began accepting applications after a notification was published in the *Federal Register* dated May 11, 1994. Applications are accepted and processed following procedures found in NIST Handbook 150.

1.4 References

1.4.1 The following documents are referenced in this handbook.

a) NIST Handbook 150, *NVLAP Procedures and General Requirements*; available from:

National Voluntary Laboratory Accreditation Program
National Institute of Standards and Technology
100 Bureau Drive, Stop 2140
Gaithersburg, MD 20899-2140

Phone: (301) 975-4016
Fax: (301) 926-2884
E-mail: nvlap@nist.gov
NVLAP Web site: http://www.nist.gov/nvlap

b) ISO/IEC/BIPM (BIPM is the Bureau International des Poids et Mesures, the International Bureau of Weights and Measures) *Guide to the Expression of Uncertainty in Measurement* (GUM), 1993.

c) ISO/IEC 17025: 1999: *General requirements for the competence of testing and calibration laboratories.*

d) ISO/IEC Guide 43: 1997, *Proficiency testing by interlaboratory comparisons, Part 1 and Part 2.*

e) ISO/IEC/BIPM *International Vocabulary of Basic and General Terms in Metrology* (VIM), 1993.

ISO documents b) through e) are available from:

Global Engineering Documents (paper copies)
Order phone: (800) 854-7179

American National Standards Institute (ANSI) (electronic copies)
Electronic Standards Store
ANSI web site: http://www.ansi.org

f) NIST Technical Note 1297, *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*. Available on-line at http://physics.nist.gov/Document/tn1297.pdf.

g) EA-2/03, EA Interlaboratory Comparison (previously EAL-P7), Mar. 1996. Available on-line at http://www.european-accreditation.org/.

h) ANSI/NCSL Z540-1-1994, *Calibration Laboratories and Measuring and Test Equipment—General Requirements*.

i) ANSI/NCSL Z540-2-1997, *U.S. Guide to the Expression of Uncertainty in Measurement*.

j) NCSL Recommended Practice RP-7: *Laboratory Design*, 1993.

k) NCSL Recommended Practice RP-11: *Reports and Certificates of Calibration*, 1991.

NCSL documents h) through k) are available from:

NCSL International
2995 Wilderness Place, Suite 107
Boulder, CO 80301-5404
Phone: (303) 440-3339
Fax: (303) 440-3384
E-mail: orders@ncsli.org
Web site: http://www.ncsli.org

l) Ehrlich, C. D., and Rasberry, S. D., "Metrological Timelines in Traceability," *J. Res. Natl. Inst. Stand. Technol.* **103**, 93 (1998).

m) Croarkin, M. C., Measurement Assurance Programs, Part II: Development and Implementation, *NBS Special Publication 676-II* (U.S. Government Printing Office, Washington, DC, 1985).

1.4.2 Additional references specific to mechanical measurements are listed in Sections 2.2 through 2.11.

1.5 Definitions

Definitions found in NIST Handbook 150 apply. However, the definitions may be interpreted differently or stated differently, when necessary to amplify or clarify the meaning of specific words or phrases as they apply to specific technical criteria.

1.5.1 Proficiency Testing: Determination of laboratory performance by means of comparing and evaluating calibrations or tests on the same or similar items or materials by two or more laboratories in accordance with predetermined conditions. For the NVLAP Calibration Laboratories Accreditation Program, this entails using a transport standard as a measurement artifact, sending it to applicant laboratories to be measured, and then comparing the applicant's results to those of a reference laboratory on the same artifact.

1.5.2 Traceability: Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties. [VIM:1993, 6.10]

A single measurement intercomparison is sufficient to establish uncertainty relationships only over a limited time interval (see reference 1.4.1 l)); internal measurement assurance (see reference 1.4.1 m)), using control (check) standards, is required to fully demonstrate that uncertainties remain within stated levels over time. For the purposes of demonstrating traceability for NVLAP accreditation, a laboratory must demonstrate not only that there is an unbroken chain of comparisons to national standards, but also that this chain is supported by appropriate uncertainties, measurement assurance processes, continuous standard maintenance, proper calibration procedures, and proper handling of standards. In this way, traceability is related to these other areas of calibration.

1.6 NVLAP documentation

1.6.1 Accreditation documents

Laboratories granted NVLAP accreditation are provided with two documents: Scope of Accreditation and Certificate of Accreditation.

The Scope of Accreditation lists the "Best Uncertainty" that an accredited laboratory can provide for a given range or nominal value within a given parameter of measurement. This "Best Uncertainty" is a statement of the smallest measurement uncertainty that a laboratory has been assessed as capable of providing for that particular range or nominal value. The actual reported value of uncertainty for any particular measurement service that the accredited laboratory provides under its scope may vary depending on such contributors as the statistics of the test and uncertainties associated with the device under test.

1.6.2 Fields of calibration/parameters selection list

The Calibration Laboratories program encompasses eight fields of physical metrology calibration, with multiple parameters under each field. Each field is covered by a separate handbook (NIST Handbooks 150-2A through 150-2H). (Fields of accreditation under Chemical Calibration are covered by separate handbooks.) Depending on the extent of its calibration capabilities, a laboratory may seek accreditation to all or only selected fields and parameters within the scope of the program. The fields of calibration and their related parameters are given on the Fields of Calibration and Parameters Selection List, which is provided to a laboratory seeking accreditation as part of the NVLAP application package for the program. Additional fields of calibration and/or parameters may be added to the Calibration Laboratories program upon request

of customer laboratories and/or if decided by NVLAP to be in the best interest of the Calibration Laboratories Program.

The laboratory is requested to indicate on the Fields of Calibration/Parameters Selection List the parameter(s) for which accreditation is desired, along with appropriate ranges and uncertainties. There is also provision for an applicant laboratory to request accreditation for parameters not currently listed on the Selection List, or for accreditation of the quality system employed for assuring Measurement and Test Equipment (M & TE) used in support of product certification. Request for accreditation of quality assurance systems for M & TE will be treated as a separate field of calibration for the purpose of setting appropriate fees. Once a laboratory meets all the requirements for accreditation for the Fields of Calibration/Parameters Selection List, this information will become the basis for the Scope of Accreditation document.

1.6.3 Checklists

Checklists enable assessors to document the assessment of the laboratory against the NVLAP requirements found in NIST Handbook 150. The NVLAP Calibration Laboratories Accreditation Program incorporates the NVLAP General Operations Checklist. The questions are applicable to evaluating a laboratory's ability to operate a calibration program, and address factors such as the laboratory's organization, management, and quality system in addition to its calibration competency.

The NVLAP General Operations Checklist is numbered to correspond to the requirements in NIST Handbook 150. Comment sheets are used by the assessor to explain deficiencies noted on the checklist. Additionally, the assessor may use the sheets to make comments on aspects of the laboratory's performance other than deficiencies.

1.7 Assessing and evaluating a laboratory

1.7.1 On-site assessment

1.7.1.1 The NVLAP lead assessor will schedule the date for on-site evaluation with the laboratory. The lead assessor will request the quality manual and documented quality and calibration procedures in advance of the visit to reduce time spent at the laboratory. Such materials will be returned by the assessor. NVLAP and the assessor will protect the confidentiality of the materials and information provided. The laboratory should be prepared to conduct routine calibrations, have equipment in good working order, and be ready for examination according to the guidance contained in this handbook, the requirements identified in NIST Handbook 150, and the laboratory's quality manual. The assessor will need time and work space to complete assessment documentation while at the laboratory. The assessor will discuss these needs at the opening meeting of the on-site assessment.

1.7.1.2 NVLAP technical assessors are provided with the NVLAP General Operations Checklist to help ensure the completeness, objectivity, and uniformity of the on-site assessment.

1.7.1.3 When accreditation has been requested for a considerable number of fields of calibration and parameters, the assessment may include observing calibrations in progress, requiring repeat measurements on completed calibrations, or listening to laboratory staff describe the calibration process. The depth of the assessment depends on the number of fields of calibration and associated parameters for which accreditation is requested and the time required to perform a given calibration.

1.7.1.4 The assessor, or the assessment team, does the following during a typical on-site assessment:

a) Conducts an entry briefing with the laboratory manager to explain the purpose of the on-site visit and to discuss the schedule for the day(s). At the discretion of the laboratory manager, other staff may attend the briefing.

b) Reviews quality system manual, equipment and maintenance records, record-keeping procedures, laboratory calibration reports, and personnel competency records. At least one laboratory staff member must be available to answer questions. However, the assessor may wish to review the documents alone. The assessor(s) does not usually ask to take any laboratory documents with him/her, and previously supplied documents will be returned.

c) Physically examines equipment and facilities, observes the demonstration of selected procedures by appropriate personnel assigned to perform calibrations, and interviews the personnel. The demonstrations can include preparation for calibration of devices, the setup and use of measuring and test equipment, standards and systems, and calculation procedures (including software) used to produce and format final results.

d) Holds an exit briefing with the laboratory manager and staff to discuss the assessment findings. Deficiencies are discussed and resolutions may be mutually agreed upon. Items that must be addressed before accreditation can be granted are emphasized, and outstanding deficiencies require response to NVLAP within 30 days. Items that have been corrected during the on-site and any recommendations are specially noted.

e) Completes an On-site Assessment Report as part of the exit briefing that summarizes the findings. The assessor(s) attaches copies of the completed checklists to this report during the exit briefing. The report is signed by the lead assessor and the laboratory's Authorized Representative to acknowledge the discussion. This signature does not necessarily indicate agreement. Challenge(s) to the findings may be made through NVLAP. A copy is given to the representative for retention. All observations made by the NVLAP assessor are held in the strictest confidence.

1.7.2 Proficiency testing

1.7.2.1 Background

Once the quality system review and on-site assessment steps have been satisfactorily completed, additional sets of data points may be gathered to aid in deciding whether or not the applicant laboratory is competent to perform calibrations within the fields of interest to the uncertainties claimed. In the eight fields of calibration covered by Handbooks 150-2A through 150-2H, there are approximately 85 parameters of interest. Under most parameters there are several subsets referred to as ranges. For example, in the mass field, parameters can range from 1 mg (10^{-6} kg) to 1 x 10^{6} kg level in value. In view of the many possible ranges, proficiency testing could be conducted in hundreds of areas. NVLAP reserves the right to test by sampling in any area. Applicant laboratories must be prepared, with reasonable notice, to demonstrate proficiency in any of a number of parameters.

1.7.2.2 Proficiency testing vs. measurement assurance

There is an important difference between proficiency testing and measurement assurance. The objective of proficiency testing is to determine through a measurement process that the laboratory's measurement results compare favorably with the measurement results of the audit laboratory (NIST or one designated by NVLAP), taking into account the relative uncertainties assigned by both the applicant and audit laboratories. The objective of proficiency testing is not to determine and certify the total measurement uncertainty of the applicant laboratory, as is done in a Measurement Assurance Program (MAP) with NIST. The objective of

proficiency testing is to verify (through the assessment process) that the uncertainty claimed by the applicant laboratory is reasonable, and then use the claimed uncertainty to test that the measurement result obtained through the proficiency test is acceptable.

It is neither the intention nor the mission of NVLAP to conduct MAPs or to otherwise provide traceability for laboratories. Laboratories obtain these services from the NIST measurement divisions. NVLAP assesses the implementation, application, and documentation of MAPs by laboratories. NVLAP accreditation encourages the use of MAPs by the calibration laboratory community, and MAP results produce objective evidence that NVLAP assessors look for as part of the assessment process.

1.7.2.3 Requirements

NVLAP's proficiency testing program uses a sampling approach. All applicant laboratories are required to complete an annual proficiency test in one parameter under each field of calibration for which accreditation has been requested. For the purposes of the NVLAP Calibration Laboratories Accreditation Program, the results of the proficiency test and the on-site visit are considered as objective evidence of a laboratory's ability to perform competent calibrations. Proficiency testing is conducted annually using different parameters in each field. However, those laboratories accredited in only one parameter within a field are retested in the same parameter.

1.7.2.4 Uncertainty determination

The applicant laboratory is required to perform a measurement or series of measurements on an artifact using the same calibration method, apparatus, and personnel that it uses to calibrate its customers' equipment. The laboratory must be able to identify and quantify all sources of uncertainty that affect the measurement. The laboratory should attach an overall uncertainty to the measurement by combining all uncertainty contributions, in their type A and type B components, in the root-sum-squared method as described in the *Guide to the Expression of Uncertainty in Measurement* (see reference 1.4.1 b)). The confidence limit used should be k = 2, which is equivalent to a 95% confidence probability.

1.7.2.5 Pass/fail criteria

The performance of the proficiency test is judged by calculating the error of the measurement, normalized with respect to the uncertainty of the measurement, using the following equation:

$$E_{normal} = \left| (Value_{lab} - Value_{ref}) / (Uncertainty_{ref}^2 + Uncertainty_{lab}^2)^{1/2} \right|$$

where

E_{normal} = normalized error of the applicant laboratory relative to the reference laboratory
$Value_{lab}$ = the value as measured by the applicant laboratory
$Value_{ref}$ = the value as measured by the reference laboratory
$Uncertainty_{ref}$ = the uncertainty of the reference laboratory
$Uncertainty_{lab}$ = the uncertainty of the applicant laboratory

To pass the proficiency test, the applicant laboratory must have a value for E_{normal} less than 1 (i.e., $E_{normal} < 1$). The results may be plotted graphically, with lines representing the limits of uncertainty of the measurements. The anonymity of each applicant laboratory will always be preserved.

1.7.2.6 Scheduling and handling

Proficiency testing is scheduled by NVLAP-designated reference laboratories. These sites are NIST laboratories or NVLAP-accredited laboratories that have the ability to perform the required proficiency tests to an uncertainty level appropriate for the laboratories they evaluate. The proficiency test is scheduled independently and does not occur at the same time as the on-site visit. Applicant laboratories are notified in advance of the approximate arrival time of the measurement artifact. Instructions for performing the test, reporting the results, communicating with the reference laboratory, and shipping are included along with the artifact as part of the proficiency test package. Applicant laboratories are instructed to perform all required measurements within a reasonable time and are told where to ship the artifacts once the testing has been completed.

1.7.2.7 Notification of results

NVLAP notifies each laboratory of its own results in a proficiency test. If a laboratory has received its on-site assessment prior to the completion of the proficiency test, the status of that laboratory's accreditation is contingent upon successful completion of proficiency testing. The laboratory's accreditation status may be changed to reflect a partial accreditation, or may be completely suspended pending demonstration of the laboratory's ability to successfully complete the proficiency test at a later date.

1.7.3 Traceability

1.7.3.1 Establishing traceability

Laboratories must establish an unbroken chain of comparisons leading to the appropriate international or national standard. The uncertainties of the comparisons must support the level of uncertainty that the laboratory gives to its customers. Generally speaking, the uncertainties of the comparisons increase as they move from a higher (international or national level) to a lower level standard. This uncertainty chain is the evidence of traceability and must be documented accordingly. Traceability does not simply mean having standards calibrated at the national laboratory. However, it must consider how a measurement, with its corresponding uncertainty, is transferred from the national level to the calibration laboratory's customers.

1.7.3.2 Considerations in determining traceability

Without some type of measurement assurance process, one cannot be reasonably certain that the comparisons have been transferred properly to the laboratory's customers. The measurement process itself must be verified to be in control over time. Therefore, traceability is not a static concept that, once established, may be ignored; it is dynamic. Process control exercised in each calibration provides the assurance that a valid transfer of the international or national standard has taken place. This assurance may be accomplished through the use of tools such as check standards and control charts. Also, the laboratory's primary standards must be maintained in such a way as to verify their integrity. Examples of this may be: (1) having more than one primary standard to use for intercomparisons; (2) monitoring the primary standard with a check or working standard (looking for changes); and (3) verifying a primary standard on a well-characterized measurement/calibration system. Using scientifically sound measurement procedures to transfer the primary standard value to the working level and the customer's item is essential to establishing traceability. If the procedure yields the wrong result, there is no way the laboratory can produce calibration data that is traceable to the international or national standard. Handling the laboratory's standards affects the measurement process, and therefore the ability to transfer the standard's value to the customer. Examples of handling problems are: (1) dirty or improperly cleaned standards; (2) maintaining standards in an improper

environment; (3) not maintaining custody and security; and (4) improper handling of standards during the measurement process.

1.7.3.3 Relationship to existing standards

The above discussion illustrates how measurement results traceability is dependent on many aspects of the measurement process and therefore must be considered in all phases of calibration. It is not coincidental that the factors addressed above are main topics of concern in ISO/IEC 17025:1999.

1.7.4 Uncertainty

NVLAP recognizes the methodology for determining uncertainty as described in the *Guide to the Expression of Uncertainty in Measurement*, published by ISO. To be NVLAP-accredited, a laboratory must document the derivation of the uncertainties that it reports to its customers. These uncertainties will appear on the scope issued to each accredited laboratory to an accuracy appropriate to the standards, procedures, and measuring devices used.

2 Criteria for accreditation

2.1 Introduction

2.1.1 Applicant laboratories are assessed using the requirements in NIST Handbook 150, *NVLAP Procedures and General Requirements*. This guide, NIST Handbook 150-2G, was developed from a NIST measurement laboratory perspective and provides examples and guidelines (not requirements) to assessors and interested calibration laboratories on good laboratory practices and recommended standards. The guide language reflects this philosophy through the use of "shoulds" instead of "shalls" (along with other less prescriptive language) when describing criteria. The requirements presented here are not absolute since specific requirements depend on the measurement uncertainty for which an applicant laboratory wishes to be accredited. This is a business decision for each laboratory and beyond the scope of NVLAP. Simply stated, to be accredited, an applicant laboratory must have a quality system and be able to prove (and document) that it is capable of doing what it says it does (i.e., correctly calibrate to a stated uncertainty) within the framework of NIST Handbook 150. Accreditation will be granted and may only be referenced in calibration reports (or other appropriate documents) for those specific parameters, ranges and uncertainties using calibration methods and procedures for which a laboratory has been evaluated. Calibrations performed by a laboratory using methods and procedures not considered appropriate for the level of measurements being made, and which have not been evaluated by the accreditation process, are outside the scope of accreditation. Such calibrations may not be referenced as "accredited" calibrations on calibration reports, etc.

2.1.2 Sections 2.2 through 2.11 detail specific calibration guidelines for mechanical measurements. This guide is dynamic in that new parameters may be added and existing criteria updated and improved.

2.2 Pressure calibration of microphones

2.2.1 Introduction

A large number of standards have been written for acoustical testing, and they represent an extremely wide variety of measurement and characterization procedures. These may involve properties of individual electroacoustical transducers and instruments, materials, and system components, as well as properties of complete systems, buildings, and test space enclosures and facilities. Because known sound pressures cannot readily be generated directly, sound pressures are usually determined by measurements using calibrated laboratory standard microphones. Consequently, this section specifically discusses the example of the commonly used pressure calibration of such microphones. However, many considerations in this example are also applicable, in varying degrees, to other acoustical test situations.

2.2.2 Scope

This section describes considerations and specific technical criteria for assessing the competence of a calibration laboratory that performs pressure calibrations of standard microphones. Some of these considerations and requirements may be applicable to other acoustical tests; but each type of acoustical testing is likely to involve some unique issues, which require the assessor to have specific knowledge of, and substantial experience with, that type of testing.

2.2.3 References

a) International Electrotechnical Commission International Standard 1094-1, *Measurement microphones. Part 1: Specifications for laboratory standard microphones*, Geneva: 1992.

b) American National Standards Institute, S1.12-1967 (R1986), *Specifications for Laboratory Standard Microphones*, currently undergoing revision, New York, NY.

c) American National Standards Institute, S1.10-1966 (R1986), *Method for Calibration of Microphones*, New York, NY.

d) International Electrotechnical Commission International Standard 1094-2, *Measurement microphones. Part 2: Primary method for pressure calibration of laboratory standard microphones by the reciprocity technique,* Geneva: 1992.

e) International Electrotechnical Commission Publication 327, *Precision method for pressure calibration of one-inch standard condenser microphones by the reciprocity technique*, Geneva: 1971.

f) International Electrotechnical Commission Publication 402, *Simplified method for pressure calibration of one-inch condenser microphones by the reciprocity technique,* Geneva: 1972.

g) International Electrotechnical Commission Publication 655, *Values for the difference between free-field and pressure sensitivity levels for one-inch standard condenser microphones*, Geneva: 1979.

h) ANSI S1.40-1984 (ASA 40-1094), *American National Standard Specification for Acoustical Calibrators*, New York: American National Standards Institute, Inc. (American Institute of Physics for the Acoustical Society of America), 1984 (R1990).

i) International Electrotechnical Commission International Standard 942, *Sound calibrators*, Geneva: 1988.

j) Nedzelnitsky, V., "Traceability of Acoustical Instrument Calibration to the National Bureau of Standards," *Proc. INTER-NOISE 80*, II, Dec. 8-10, 1980, Miami, FL, G. C. Maling, Jr., Ed., Poughkeepsie, NY: Noise Control Foundation, 1043 (1980).

k) Nedzelnitsky, V., Burnett, E. D., and Penzes, W. B., "Calibration of Laboratory Condenser Microphones," *Proceedings of the 10th Transducer Workshop*, Transducer Committee, Telemetry Group, Range Commanders Council, Colorado Springs, CO (June 1979).

l) Nedzelnitsky, V., "Laboratory Microphone Calibration Methods at the National Institute of Standards and Technology," in: *AIP Handbook of Condenser Microphones: Theory, Calibration, and Measurements*, G. S. K. Wong and T. F. W. Embleton, Eds., American Institute of Physics Press, New York, 1994.

m) Koidan, W., "Calibration of Standard Condenser Microphones: Coupler Versus Electrostatic Actuator," *J. Acoust. Soc. Am.*, **44**, (5), 1451 (Nov. 1968).

n) Cook, R. K., Edelman, S., and Koidan, W., "Calibrations of Microphones, Vibration Pickups, and Earphones," *J. Audio Eng. Soc.*, **13**, (4), (Oct. 1965).

o) Koidan, W., and Siegel, D. S., "Free-Field Correction for Condenser Microphones," *J. Acoust. Soc. Am.,* **36**, (11), 2233 (Nov. 1964).

p) Koidan, W., "Hydrogen Retention System for Pressure Calibration of Microphones in Small Couplers," *J. Acoust. Soc. Am.*, **35**, (4), 614 (Apr. 1963).

q) Koidan, W., "Method of Measurement of | E'/I' | in the Reciprocity Calibration of Condenser Microphones," *J. Acoust. Soc. Am.*, **32**, (5), 611 (May 1960).

2.2.4 Accommodation and environmental conditions

2.2.4.1 Electronic and other instruments should be turned on for a sufficiently long period before beginning calibration that they reach equilibrium. For example, any warmup time necessary for an instrument should be provided. The microphones and all other apparatus should be allowed to reach room temperature prior to being calibrated. During calibration, the temperature should be monitored close to the microphones.

2.2.4.2 Recommended environmental conditions during calibrations are:

a) 23 +3, - 5 °C temperature,

b) 101.325 +1, -3 kPa barometric pressure, and

c) 50 +25, -35 percent relative humidity.

2.2.4.3 Ambient acoustical noise and vibration in the laboratory should be sufficiently low so that the guidelines in this document are met. For example, the signal to noise ratios during measurements should be sufficiently high so that the claimed uncertainties of calibration can be achieved.

2.2.5 Equipment

2.2.5.1 Microphones used as primary reference standards should be calibrated sufficiently often that the microphones' sensitivity level does not change between calibrations by more than the corresponding allowance for such change in the uncertainty component for the reference standard microphone in the laboratory's estimate of its overall uncertainties. Typically, calibration of reference laboratory standard microphones should be performed annually. If damage to, or instability of, these microphones is suspected, more frequent calibration is indicated to resolve this problem.

2.2.5.2 Microphones used as secondary standards should be calibrated sufficiently often that the microphones' sensitivity level does not change between calibrations by more than the corresponding allowance for such change in the uncertainty component in the applicant's estimate of its overall uncertainties of calibrations in which these microphones serve as secondary standards. Typically, calibration of secondary standard microphones should be performed annually. If damage to, or instability of, these microphones is suspected, more frequent calibration is indicated to resolve this problem.

2.2.6 Test and calibration methods and method validation

2.2.6.1 A primary calibration of a microphone determines the pressure response level (in decibels, with reference: one volt/pascal), also known as the pressure sensitivity level, by using a method in which no calibrated microphone of known response level is required. ANSI S1.10, IEC 1094-2, and its predecessors IEC 327 and IEC 402 describe variations of the reciprocity method in essentially closed acoustical couplers. This method achieves primary pressure calibration of microphones by a series of electrical and mechanical measurements, including measurements of barometric pressure, temperature, and relative humidity, as well as knowledge of properties of the gas filling the couplers, the volumes of the couplers, etc. Microphones that have received primary pressure calibration are typically used as primary reference standards to calibrate other microphones. These microphones, or sometimes even the reference microphones themselves, are used to

calibrate acoustical instruments such as sound calibrators, audiometers, etc., that require the measurement of sound pressure.

2.2.6.2 Microphones used as primary reference standards are typically used to calibrate other microphones by comparison methods that may be substitution methods, as described in ANSI S1.10-1966 (R1986). Somewhat more accurate and less laborious reciprocity-based comparison methods may also be used as discussed in the above references by Nedzelnitsky, Burnett, and Penzes, and by Nedzelnitsky. These other microphones are then typically used as secondary standard (working standard) microphones to calibrate other instruments. Such instruments include acoustical or sound calibrators specified in ANSI S1.40-1984 (ASA 40-1094) and IEC 942, audiometers, other microphones and microphone systems, etc.

2.2.7 Handling of test and calibration items

Properly calibrated laboratory standard microphones, as defined and specified in ANSI S1.12-1967 (R1997) and IEC 1094-1, as well as other standard microphone configurations such as those specified in IEC 655, can be very stable and accurate instruments when used and stored under laboratory conditions. However, these microphones should not be exposed to corrosive conditions. They also should be protected from mechanical shock, from rapid changes in environmental conditions which may cause condensation, and from rapid changes in temperature which may cause thermal shock. Because the diaphragms of such microphones are extremely fragile, responsible personnel should always exercise proper care in handling them.

2.2.8 Records

All measurements should be recorded. The data sheets or the computer file, or both, containing the data should include the model and serial number of the microphone being calibrated, the date of calibration, and other pertinent information. Records of calibrations should be retained for a period of time specified in the quality manual. Records associated with primary and secondary standard microphones should be kept for the entire lifetime of those microphones.

2.2.9 Reporting the results

The laboratory should be responsible for providing calibration reports that document measurement results that are within the uncertainty estimates. In addition to the reporting requirements in ISO/IEC 17025, the calibration report should also contain the following information:

a) manufacturer, model, and serial number of each microphone calibrated,

b) brief description of the method of calibration,

c) for comparison calibrations, the type (including manufacturer and model) of reference microphone(s) used and the origin of its (their) calibration(s),

d) barometric pressure, temperature, and relative humidity at which the calibration was performed,

e) the frequency range of calibration for which the laboratory has been accredited should be clearly and prominently stated.

2.2.10 Considerations in determining the adequacy of primary calibration

2.2.10.1 In principle, a laboratory performing primary calibrations of its own microphones can demonstrate the adequacy of these calibrations by either of two approaches.

a) Under the first approach, the laboratory seeking accreditation (henceforth termed the applicant) should provide descriptions of the primary calibration method and its uncertainty estimate (including the necessary individual uncertainty components, the manner in which they have been determined, and the manner in which they are combined to arrive at this estimate). This information should be sufficiently detailed to allow the assessor to establish the adequacy of laboratory procedures and their consistency with this estimate. This approach will probably require the provision of information regarding the manufacturer, model, specifications, and other performance data, including records of calibration (ideally performed at regular, periodic intervals, but in any event commensurate with the instrument characteristics and the necessary uncertainty), of all instruments upon which the primary calibration method depends. This approach requires that the applicant demonstrate that its calibration is performed by a recognized (e.g., standardized) primary calibration method, with its component methods traceable to other instrument properties and calibrations, fundamental physical properties and constants, etc.

b) In the other approach, the applicant should provide an estimate of its calibration uncertainty and a record of interlaboratory testing in which microphone calibrations performed by the applicant at regularly scheduled, periodic time intervals are compared with calibrations performed at NIST or a laboratory designated by NVLAP. This approach, which should also consider the stability of the particular microphone(s) used, effectively comprises evaluation of results of a measurement assurance program.

2.2.10.2 In practice, an applicant often can provide some information and data from each approach. The data from only one approach is often insufficient to allow the assessor to judge the validity of the uncertainty estimate. In such cases, the assessor should judge whether the sum of all the available information and data is sufficient to support the uncertainty estimate supplied by the applicant.

2.2.10.3 Specific examples of measured, or otherwise determined, quantities and properties that contribute uncertainty components to a primary pressure calibration by the reciprocity method include

a) the geometrical volumes of the acoustical couplers used,

b) the (sine wave signal) frequency-dependent equivalent volumes of the microphones,

c) the (sine wave signal) frequency-dependent corrections for the effects of acoustical wave motion, of slight departures from adiabatic sound propagation in these couplers, and of the acoustical influence of capillary tubes (needed to equalize static pressures inside and outside the coupler),

d) frequency of calibration,

e) electrical transfer impedances,

f) dc polarizing voltages applied to the microphones,

g) ambient static (barometric) pressure, temperature, relative humidity, and

h) the values of thermodynamic properties such as the ratio(s) of specific heats of the gas(es) used to fill the coupler(s).

2.2.10.4 Other, less directly obvious, issues that can affect the uncertainty of calibration include:

a) the possible influence of the relative rotational positions of the microphones in the coupler(s),

b) the signal-to-noise ratios during measurements, and crosstalk between the relatively large voltages at the electrical terminals of the reciprocal microphone used as a source, at the input to an attenuator during an insert voltage measurement, or following the outputs of amplification stages in the measurement system, and

c) the relatively small voltages at the input to the receiving microphone preamplifier.

2.2.10.5 Since these calibrations are almost always performed with sine wave signals, and the calibration uncertainties are generally dependent upon the signal frequency, the frequency range for which accreditation has been granted should be specified.

2.2.11 Considerations in determining the adequacy of secondary calibration

2.2.11.1 An applicant performing secondary calibrations should demonstrate the adequacy of the calibrations of microphones used as primary reference standards. If the applicant calibrates these primary standards, it should demonstrate this adequacy as discussed in Sections 2.2.5.1 and 2.2.10. If the applicant does not calibrate these primary standards, the applicant should provide records including formal test reports or certificates demonstrating that these standards receive calibrations at regularly scheduled, periodic time intervals by NIST, or by an accredited laboratory.

2.2.11.2 The applicant should also demonstrate the adequacy of the secondary calibrations. In principle, this can be accomplished by either of two approaches.

a) In the first approach, the applicant should provide descriptions of the secondary calibration method and its uncertainty estimate (including the necessary individual uncertainty components, the manner in which they have been determined, and the manner in which they are combined to arrive at this estimate). This information should be sufficiently detailed to allow the assessor to establish the adequacy of laboratory procedures and their consistency with this estimate. This approach will probably require provision of information regarding the manufacturer, model, specifications, and other performance data, including records of calibration (preferably performed at regularly scheduled, and, ideally, at periodic, intervals, but in any event commensurate with the necessary uncertainty), of all instruments upon which the secondary calibration method depends. This approach requires that the applicant demonstrate that calibration is performed by a recognized secondary calibration method, with its component methods traceable to other instrument properties and calibrations, etc.

b) In the other approach, the applicant should provide an estimate of its secondary calibration uncertainty and a record of interlaboratory testing in which secondary microphone calibrations performed by the applicant at regularly scheduled, periodic time intervals are compared with calibrations performed by NIST or by an accredited laboratory. This approach effectively comprises evaluation of the results of a measurement assurance program.

2.2.11.3 In practice, an applicant often can provide some information and data from each approach. The data from only one approach is often insufficient to allow the assessor to validate the uncertainty estimate. In such

cases, the assessor should judge whether the sum of all the available information and data is sufficient to support the uncertainty estimate supplied by the applicant.

2.2.11.4 The assessor should consider specific examples of measured or otherwise determined quantities that contribute uncertainty components to a secondary pressure calibration by the comparison or reciprocity-based comparison methods. Depending upon the methods, these might include: the geometrical volumes of the acoustical couplers used, the (sine wave signal) frequency-dependent equivalent volumes of the microphones, frequency of calibration, ac electrical voltages or voltage ratios, dc polarizing voltages applied to the microphones, ambient static (barometric) pressure, temperature, and relative humidity. Other, less obvious issues that affect the uncertainty of calibration include: the signal-to-noise ratios during measurements, and crosstalk between the relatively large voltages at the electrical terminals of the reciprocal microphone or other transducer used as a source, at the input to an attenuator during an insert voltage measurement, or following the outputs of amplification stages in the measurement system, and the relatively small voltages at the input to the receiving microphone preamplifier.

2.2.11.5 Since these calibrations are almost always performed with sine wave signals, and the calibration uncertainties are generally dependent upon the signal frequency, the frequency range for which accreditation has been granted should be specified.

2.3 Accelerometer calibrations

2.3.1 Background

Calibrations of accelerometer sensitivity can be performed over a variety of frequency and amplitude ranges using a number of different (i.e., independent) methods. The accelerometer to be calibrated could be intended by design for use as a primary standard, secondary standard, or field instrument. Ideally, any system employed to calibrate the sensitivity of an accelerometer by comparison within a given range of amplitudes and frequencies should ultimately be traceable to a primary standard. That primary standard should be calibrated over the same range of amplitudes and frequencies using measurements of base-unit quantities to determine the absolute sensitivity of the primary standard.

2.3.2 Scope

This section is restricted to frequency-domain calibrations of the magnitude of axial sensitivity of rectilinear accelerometers using either discrete sinusoidal or random noise excitation by either absolute or comparison methods. For the purposes of this section, a standard accelerometer may be considered to consist of a transducer (typically a piezoelectric or piezoresistive device), a transducer in combination with a signal conditioning amplifier or integral electronics, or a transducer in combination with a signal conditioning amplifier, or integral electronics, and the moving element of a vibration exciter. The calibration of four different types of standards are considered and defined in this guide: primary, transfer, secondary, and working. Laboratories with the capability of achieving the greatest calibration uncertainty will maintain either primary or transfer standards with a history of repeated absolute calibrations.

2.3.3 References

a) ANSI S2.2-1959 (R 1990), *American national standard methods for the calibration of shock and vibration pickups*, American Standards Association, Inc., New York, NY, 1960.

b) ANSI S2.11-1969 (R 1986), *American national standard for the selection of calibrations and tests for electrical transducers used for measuring shock and vibration*, American Standards Association, Inc., New York, NY, 1970.

c) ISO/DIS 5347 1993. International Organization for Standards, *Draft international standard methods for the calibration of vibration and shock pick-ups*. Available from: American National Standards Institute, Inc., New York, NY.

d) ANSI S2.61-1989 (R 1991) (ASA 78), *American national standard guide to the mechanical mounting of accelerometers*, American National Standards Institute, Inc. (American Institute of Physics for the Acoustical Society of America), New York, NY, 1989.

e) Robinson, D. C.; Serbyn, M. R.; Payne, B. F., "A description of NBS calibration services in mechanical vibration and shock," *Nat. Bur. Stand. (U.S.) Tech. Note 1232*; 1987 February.

f) Levy, S.; Bouche, R. R., "Calibration of vibration pickups by the reciprocity method," *J. Res. Nat. Bur. Stand.* (U.S.) **57**, (4): 227-243; 1956 October.

g) Dimoff, T., "Electrodynamic vibration standard with a ceramic moving element," *J. Acoust. Soc. Am.* **40**, (3): 671-676; 1966 September.

h) Payne, B. F., "An automated fringe counting laser interferometer for low frequency vibration measurements," *Proceedings of the 32th International Instrumentation Symposium*, 1986 May; Seattle, WA (U.S.). Research Triangle Park, NC: Instrument Soc. Am.; 1986. Paper no.: 86-0101.

i) Payne, B. F.; Serbyn, M. R., "An application of parameter estimation theory in low frequency accelerometer calibrations," *Proceedings of the 14th Transducer Workshop*, 1987 June 16-18; Colorado Springs, CO. White Sands Missile Range, NM: Secretariat Range Commanders Council; 1987: 162-170.

j) Schmidt, V. A.; Edelman, S; Smith, E. R.; Pierce, E. T., "Modulated photoelectric measurement of vibration," *J. Acoust. Soc. Am.*, **34**, (4): 455-458; 1962 April.

k) Payne, B. F., "Automation of vibration testing at the National Bureau of Standards," *Proceedings of the 30th Annual Technical Meeting of the Institute of Environmental Sciences*, 1984 May 1-3; Orlando, FL. Mt. Prospect, IL: IES; c1984: 478-482.

l) Payne, B. F., "The application of back-to-back accelerometers to precision vibration measurements," *J. Res. Nat. Bur. Stand.* (U.S.), **88**, (3): 171-174; 1983 May-June.

2.3.4 Definitions

2.3.4.1 Absolute calibration: A calibration process in which an instrument is calibrated by deriving absolute sensitivity from measurements of quantities contained in a base system of units.

2.3.4.2 Comparison calibration: A calibration process in which an instrument is calibrated by comparison of its output with that of another instrument, usually one with an absolute calibration.

2.3.4.3 Primary standard: A laboratory standard maintained and validated at a given laboratory with a history of repeated absolute calibrations.

2.3.4.4 Secondary standard: A laboratory standard maintained and calibrated at a given laboratory with a history of repeated comparison calibrations.

2.3.4.5 Sensitivity: The characteristic electrical output of a standard accelerometer per unit acceleration.

2.3.4.6 Transfer standard: A laboratory standard with a history of repeated absolute or comparison calibrations performed at a primary standards laboratory different from that maintaining it.

2.3.4.7 Working standard: A laboratory standard, usually calibrated by comparison against a primary or transfer standard, with a history of repeated calibrations that is used to perform a relatively large number of comparison calibrations of lesser uncertainty.

2.3.5 Accommodation and environmental conditions

2.3.5.1 Typically, accelerometers are relatively impervious to small changes in environmental conditions. However, they can be damaged or their performance permanently altered by exposure to large temperature extremes. Accelerometers can also be damaged by exposure to mechanical signals with vibration levels larger than those imposed by the physical limitations of the accelerometer. Prior to use, accelerometers should be allowed to stabilize under ambient test conditions. It is recommended that the temperature of the laboratory be controlled to approximately 23 °C and that the relative humidity not exceed 55 percent. The calibration laboratory should exhibit relatively small levels of airborne and structural noise. In particular, the vibration isolation of any exciter used in the calibration of accelerometers should be demonstrably adequate for the purposes of the measurements performed. Any standard should be calibrated under the same nominal ambient conditions as those present when in use.

2.3.5.2 Electrical power should be conditioned and measurement systems should be grounded in such a way as to provide adequate ratios of signal to noise for the purposes of the measurements performed. Coaxial cables connected to accelerometers should be secured in such a way as to minimize triboelectric effects and effects from cable strain.

2.3.6 Equipment

2.3.6.1 Reference standards (primary, transfer, or both) maintained at the accredited laboratory should be validated or calibrated periodically by NIST, or an accredited laboratory maintaining primary vibration standards, on a time interval appropriate to the uncertainty statement associated with the long-term stability, calibration, and use of these standards. The accredited laboratory should maintain at least one transfer standard. That transfer standard should be periodically calibrated on a statistically appropriate time interval at either NIST, or an accredited laboratory maintaining primary vibration standards. That laboratory should not be the laboratory maintaining the transfer standard. Any accredited laboratory that claims primary calibration capability should maintain at least one transfer standard that is periodically calibrated on a statistically appropriate time interval at NIST. Prior to accreditation, the laboratory should demonstrate, by a statistically suitable number of calibrations of the same transfer standard performed at NIST and the laboratory under accreditation, that reference standards and calibration systems can be used and maintained at the laboratory under accreditation within the stated statistical estimates of uncertainty.

2.3.6.2 Laboratory standards (secondary, working, or both) calibrated and maintained at the accredited laboratory should be calibrated periodically on a time interval appropriate to the uncertainty statement associated with the long-term stability, calibration, and use of these standards.

2.3.6.3 Instruments that are critical to maintaining the uncertainty associated with systems of calibration, and that are relied upon to measure absolute or relative quantities, or maintain a standard value, e.g., standard capacitors, resistors, etc., should be calibrated by either NIST, or an accredited laboratory maintaining suitable primary standards, on time intervals that are appropriate to the long-term stability of these instruments. The amplitude and frequency ranges over which these instruments are calibrated should include the amplitudes and frequencies typical of the signals that the instruments encounter when in use. If the calibration system relies on calibration by comparison, then the calibration of the standards utilized by the system should be traceable to a standard with a primary calibration over ranges of amplitude and frequency that includes those amplitudes and frequencies produced by the calibration system during use.

2.3.7 Records

2.3.7.1 Documentation on the calibration of all standards and calibration-system instruments used in the accredited laboratory should be retained for an indefinite period of time.

2.3.7.2 Records should be made that document the specific instruments included in a calibration system each time that system is used to perform a calibration.

2.3.7.3 Any time that an instrument or measurement procedure is changed in a calibration system, the performance of the altered calibration system should be verified, and the performance verification should be adequately documented.

2.3.8 Reporting the results

2.3.8.1 In addition to the reporting requirements in ISO/IEC 17025, calibration reports should state sensitivity as a function of frequency, and at each frequency reported should include the approximate amplitude of excitation during calibration.

2.3.8.2 A statement of estimated uncertainty should be given either as a worst-case number, or as function of frequency, over the entire range of frequencies reported.

2.3.8.3 Further, calibration reports should include

a) the manufacturer, model (type), and serial numbers of all instrumentation included in the unit calibrated,

b) any instrument settings of the unit when calibrated,

c) the mounting torque used to attach the test transducer to the vibration exciter,

d) the condition of the mating surfaces of the test transducer and vibration exciter, and

e) the environmental conditions during calibration if these are significantly different from those recommended above (see 2.3.5).

2.4 Air speed calibrations

2.4.1 Scope

2.4.1.1 This section contains specific technical criteria which a laboratory should meet if it is to be recognized as competent to carry out calibrations on air speed artifacts.

2.4.1.2 The artifact calibrations currently included in the accreditation program are:

a) pitot-static tubes,

b) hot wire anemometers,

c) hot film anemometers,

d) cup anemometers,

e) propeller anemometers,

f) ultrasonic anemometers,

g) velocity grids,

h) velocity probes, and

i) vane anemometers.

2.4.1.3 The artifact calibration facilities currently included in the accreditation program are:

a) high-velocity wind tunnel for meteorological-type anemometers, and

b) low-velocity wind tunnel using Laser Doppler Velocimetry (LDV) such as used for indoor air quality-type anemometers.

2.4.2 References

a) NIST SP 250, "NIST Calibration Services Users Guide," available on line at http://ts.nist.gov/ts/htdocs/230/233/calibrations/.

b) Mease, N. E., Cleveland, Jr., W. G., Mattingly, G. E., and Hall, J. M., "Air Speed Calibrations at the National Institute of Standards and Technology," *Proceedings of the 1992 Measurement Science Conference*, pp 315-326, Anaheim, CA, 1992, available on-line at http://www.cstl.nist.gov/div836/836.01/PDFs/1992/MSC_1992_Air_Speed.pdf.

c) Bean, V. E., Hall, J. M., "New Primary Standards for Air Speed Measurement at NIST," *Proceedings of the 1999 NCSL Workshop and Symposium* (Charlotte, NC: NCSL 1999) available on-line at http://www.cstl.nist.gov/div836/836.01/PDFs/1999/NCSL_055.pdf.

2.4.3 Areas of commonality among all parameters

2.4.3.1 Assuring the quality of test and calibration results

a) All sources of air speed measurement variability for the calibration should be monitored (e.g., thermometry, pressure instrumentation calibration, humidity measuring devices, etc.) and check standards should be used to ensure that the calibrations are carried out under controlled conditions. The laboratory should maintain statistical process control (SPC) commensurate with the uncertainty levels needed for the calibration. The SPC control parameters should be based on measurements of check standards (or closure parameters), the imprecision of which can be quantified using multiple measurements. Type B uncertainties of the measurement process should be quantified using the true value of the result or an estimate of it. The frequency and number of process control checks should be appropriate for the level of uncertainty claimed for the calibration.

b) The laboratory should have control artifacts which adequately span the range of materials and parameters normally calibrated by the laboratory. Every observed value of each control should be recorded and should be compared to its historic value to determine whether or not the process is in control. These values should be plotted on a control chart (may be done on a computer and stored electronically) that has upper and lower control limits.

2.4.3.2 Accommodation and environmental conditions

a) The environmental conditions (e.g., temperatures, pressures, relative humidity, etc.) in the air speed calibration area should be controlled and measured, with maximum variations not exceeding those permitted by the materials and the uncertainty level needed for the calibration.

b) The environmental conditions (e.g., temperatures, pressures, relative humidity, etc.) in the air speed calibration facility (a wind tunnel or similar testing facility) should be controlled and measured, with maximum variations not exceeding those permitted by the materials and the uncertainty level needed for the calibration.

2.4.3.3 Equipment

a) The laboratory should have the equipment needed to make auxiliary measurements on pertinent metering assemblies or artifacts (e.g., wind tunnel or similar testing facility, and the measurement techniques and associated instrumentation needed for such a testing facility).

b) The laboratory should have atmospheric-condition measuring capabilities suitable to the calibration procedure (e.g., measurement of the barometric pressure, wind-tunnel temperature, ambient temperature, relative humidity, etc.).

c) A laboratory that certifies artifacts to specified tolerances should demonstrate a measurement uncertainty which does not exceed 50 percent of the tolerance. Exceptions will be accepted for measurement systems which are documented to be state-of-the-art.

2.4.3.4 Handling of test and calibration items

a) Air speed artifacts should be inspected for deficiencies which could otherwise damage both the performance of the artifact and the testing facility.

b) Air speed artifacts should be cleaned and stored in a manner to prevent accidental contact with materials which could cause damage to them.

c) Air speed artifacts should not be used above their wind speed limits.

2.4.3.5 Reporting the results

The uncertainty for the measurement results reported should be derived from a model of the measurement system which includes, as applicable, the uncertainties due to:

a) master artifact,

b) long term reproducibility of measurement system,

c) statistics for multiple measurements at a particular air speed,

d) environmental conditions, and

e) other factors, as appropriate.

2.4.4 Unique areas of consideration

2.4.4.1 Pitot-static tube calibration

2.4.4.1.1 Scope

This section sets out some specific technical requirements for a laboratory to be recognized as competent to carry out Pitot-static tube calibrations.

2.4.4.1.2 References

a) Goldstein, Richard J.,editor, *Fluid Mechanics Measurements*, Hemisphere Publ. Co., New York, NY, 1979.

b) Bean, V. E., Hall, J. M. "New Primary Standards for Air Speed Measurement at NIST," *Proceedings of the 1999 NCSL Workshop and Symposium* (Charlotte, NC: NCSL 1999).

c) Bean, V. E., Hall, J. M., and Mattingly, G. E., "Preliminary Results from Interlaboratory Comparison of Air Speed Measurements Between 0.3 m/s and 15 m/s," Proceedings of the 1999 NCSL Workshop and Symposium (Charlotte, NC: NCSL 1999).

d) Mease, N. E., Cleveland, W. G., Jr., Mattingly, G. E., and Hall, J. M., "Air Speed Calibrations at the National Institute of Standards and Technology," *Proceedings of the 1992 Measurement Science Conference* (Anaheim, CA: MSC 1992), pp. 315-326.

2.4.4.1.3 Assuring the quality of test and calibration results

The laboratory should have a laboratory-standard Pitot-static tube, the data from which is directly traceable to NIST, and the capability to accurately measure the differential pressure produced between the total pressure and static pressure ports for both the laboratory standard and the instrument under test. The

laboratory-standard Pitot-static tube should be calibrated at least every other year, and the historical data should be used to establish and demonstrate statistical process control.

2.4.4.1.4 Accommodation and environmental conditions

The laboratory should accurately monitor the environmental conditions of both the measuring area and inside the wind tunnel or similar testing facility. Also, the laboratory should document and maintain historical reference data regarding the performance of any instrumentation utilized in the calibration of Pitot-static tubes.

2.4.4.1.5 Equipment

a) The laboratory should have the equipment needed to make air speed measurements for both the laboratory-standard Pitot-static tube and the tube under test.

b) The laboratory should determine the uncertainty of its air speed measurements through periodic artifact calibration and statistical and process control of the testing facility referenced to accepted methods and practices (e.g., Bernoulli's Law as a way to determine air speed using the measured differential pressure between the total pressure and static pressure ports of a Pitot-static tube).

2.4.4.1.6 Test and calibration methods and method validation

a) The laboratory should have a manual outlining the procedures to be followed for each type of calibration. For Pitot-static tubes, it should detail:

   1) methods to determine if the laboratory standard tube and the tube under test are in proper working order,

   2) the positioning of each tube in the testing apparatus,

   3) procedures for the actual calibration, and

   4) procedures for data reduction and analysis.

b) Calibration methods should be used which identify the performance criteria of the laboratory standard vs. the tube under test. These methods should not, in and of themselves, create situations which will affect the performance, such as:

   1) interference caused by improper separation of devices,

   2) differences in particular measuring devices, and

   3) improper placement in the testing apparatus.

2.4.4.1.7 Handling of test and calibration items

a) Before items are calibrated, they should be visually inspected and tested to insure that they are in proper working order. Pitot-static tubes should be tested for leaks using accepted methods.

b) When installing Pitot-static tubes in a testing device, methods should be used which will insure maximum performance of the testing facility and the tube and which will not cause damage to, or restriction of, the tube.

2.4.4.1.8 Reporting the results

a) The reported uncertainty of Pitot-static tube calibrations should be derived from a model of the measurement system which includes, as applicable, uncertainties due to:

   1) environmental conditions (i.e., ambient temperature, barometric pressure, and relative humidity, etc.),

   2) test conditions (i.e., stagnation temperature, stagnation pressure, relative humidity, etc.),

   3) differential pressure of Pitot-static tubes,

   4) control of speed of testing device,

   5) set-up and actual test,

   6) NIST calibration, and

   7) other factors, as appropriate.

b) In addition to general requirements of ISO/IEC 17025, reports should state:

   1) testing facility information such as turbulence level, boundary layer thickness, size of facility, etc.,

   2) performance of Pitot-static tube under test,

   3) environmental conditions, and

   4) other factors, as appropriate.

2.4.4.2 Low-velocity airflow facility using Laser Doppler Velocimetry

2.4.4.2.1 Scope

This section sets out specific technical requirements for a laboratory to be recognized as competent to perform low-velocity air flow calibrations using Laser Doppler Velocimetry (LDV).

2.4.4.2.2 A low-velocity air flow calibration facility should be calibrated directly, by measuring such controlled parameters as laser wavelength and the crossing angle of the beam. However, the recommended procedure for certification, artifact traceability to NIST, requires a mobile artifact, (for example, a Pitot-static tube).

2.4.4.2.3 A low-velocity air flow facility should generate accurate measurements of air speed in a range where, typically, a Pitot-static tube cannot meet the desired uncertainty requirements. The higher-speed range of the facility should include velocities at which the performance of a Pitot-static tube meets the desired uncertainty requirements, i.e, 5 m/s and above.

2.4.4.2.4 LDV is a method which provides certain advantages when used in a low-velocity air flow facility. For example:

a) it is nonintrusive;

b) it has a linear response with air speed which can be derived from first principles; and,

c) it has a very wide air speed range.

2.4.4.2.5 The artifact calibrations currently included in the accreditation program are:

a) hot-wire anemometers,

b) hot-film anemometers,

c) velocity grids,

d) velocity probes,

e) low-speed vane anemometers,

f) ultrasonic anemometers, and

g) other air speed instrumentation.

2.4.4.2.6 References

a) Purtell, L. P., and Klebanoff, P. S., "A Low-Velocity Airflow Calibration and Research Facility," *NBS Tech Note 989* (March 1979).

b) Goldstein, Richard J., editor, *Fluid Mechanics Measurements*, Hemisphere Publ. Co., New York, NY, 1979.

c) Bean, V. E., Hall, J. M. "New Primary Standards for Air Speed Measurement at NIST," *Proceedings of the 1999 NCSL Workshop and Symposium* (Charlotte, NC: NCSL 1999).

d) Bean, V. E., Hall, J. M., and Mattingly, G. E., "Preliminary Results from Interlaboratory Comparison of Air Speed Measurements Between 0.3 m/s and 15 m/s," Proceedings of the 1999 NCSL Workshop and Symposium (Charlotte, NC: NCSL 1999).

e) Mease, N. E., Cleveland, W. G., Jr., Mattingly, G. E., and Hall, J. M., "Air Speed Calibrations at the National Institute of Standards and Technology," *Proceedings of the 1992 Measurement Science Conference* (Anaheim, CA: MSC 1992), pp. 315-326.

2.4.4.2.7 Equipment

a) The laboratory should have an LDV system in a wind tunnel or similar testing facility.

b) A suitable airflow facility should meet certain criteria. The facility should:

1) avoid blocking effects,

2) have good speed control,

3) provide an air stream with spatial uniformity and low turbulence, and

4) monitor environmental conditions.

c) The laboratory should have the equipment needed to make air speed measurements for both the laboratory-standard Pitot-static tube and the LDV system.

2.4.4.2.8 Assuring the quality of test and calibration results

a) The LDV system should be calibrated directly or calibrated using a laboratory-standard Pitot-static tube, directly traceable to NIST. The calibration of the LDV using the laboratory-standard Pitot-static tube should be done periodically, with the resulting data used for process control.

b) Calibration of the LDV should be done using an appropriate laboratory standard, such as a Pitot-static tube, at velocities which produce a sufficiently large pressure differential in the tube that the uncertainty in the pressure measurement does not propagate an inappropriately large error into the air speed determination, i.e., at or above 5 m/s.

2.4.4.2.9 Accommodation and environmental conditions

The laboratory should monitor the environmental conditions of the measuring area and in the test section of the wind tunnel or similar testing device. Also, the laboratory should document and maintain historical reference data regarding the performance of any instrumentation used in calibrations using LDV.

2.4.4.2.10 Test and calibration methods and method validation

a) The laboratory should have a manual outlining the procedures to be followed for each type of calibration. For LDV measurement calibrations, important considerations would include:

1) procedures for performing the actual calibration,

2) positioning of the instrument under test,

3) determining if the LDV system is calibrated and in control, and

4) placement of the laser sensing volume in the testing facility.

b) Other calibration methods, including data reduction and analysis, should be detailed.

c) Calibration methods should be used that identify the performance criteria of the instrument under test and that do not, in and of themselves, create situations which affect the performance.

2.4.4.2.11 Handling of test and calibration items

a) Before items are calibrated, they should be visually inspected and tested to insure they are in proper working order.

b When installing air speed instrumentation in a testing facility, methods should be used which will insure satisfactory performance of the testing facility and of the instrument under test.

2.4.4.2.12 Reporting the results

a) The laboratory should determine the uncertainty of its air speed measurements through periodic calibrations performed by NIST and statistical and process control of the testing facility referenced to accepted methods and practices.

b) The reported uncertainty of low-velocity airflow calibrations using LDV should be derived from a model of the measurement system that includes, as applicable, uncertainties due to:

1) environmental conditions (i.e., ambient temperature, barometric pressure, and relative humidity, etc.),

2) test conditions (i.e., stagnation temperature, stagnation pressure, relative humidity, etc.),

3) differential pressure of Pitot-static tubes,

4) control of speed of testing device,

5) set-up and actual test,

6) NIST calibration uncertainties, and

7) other factors, as appropriate.

c) Reports and certification should be provided in accordance with ISO/IEC 17025 requirements. They should state:

1) laboratory uncertainty,

2) ay applicable traceability information,

3) laboratory calibration method,

4) evironmental conditions, and

5) other factors, as appropriate.

2.4.4.3 Air speed measurements for meteorological-type anemometers

2.4.4.3.1 This section sets out specific requirements for a laboratory to be recognized as competent to carry out air speed calibrations for meteorological-type anemometry.

2.4.4.3.2 A facility for calibrating meteorological-type anemometers should be a wind tunnel or similar device which can accurately attain air speeds adequate to the requirements of the industry. Meteorological-type air speed instrumentation measuring air speeds up to 50 m/s (approx. 100 mph) are not uncommon, although the top speed of many instruments of this type may be not more than 25 m/s (approx. 50 mph).

2.4.4.3.3 The facility should have an adequately large test area, since the types of instrumentation typically used can be larger and much bulkier than either Pitot-static tubes or low-velocity air speed instruments such as hot-wire anemometers.

2.4.4.3.4 The artifact calibrations currently included in the accreditation program are:

a) hot-wire anemometers,

b) hot-film anemometers,

c) velocity probes,

d) high-speed vane anemometers,

e) propeller anemometers,

f) ultrasonic anemometers,

g) pitot-static tubes, and

h) cup anemometers.

2.4.4.3.5 References

a) Goldstein, Richard J., editor, *Fluid Mechanics Measurements*, Hemisphere Publ. Co., New York, NY, 1979.

b) Bean, V. E., Hall, J. M. "New Primary Standards for Air Speed Measurement at NIST," *Proceedings of the 1999 NCSL Workshop and Symposium* (Charlotte, NC: NCSL 1999).

c) Bean, V. E., Hall, J. M., and Mattingly, G. E., "Preliminary Results from Interlaboratory Comparison of Air Speed Measurements Between 0.3 m/s and 15 m/s," Proceedings of the 1999 NCSL Workshop and Symposium (Charlotte, NC: NCSL 1999).

d) Mease, N. E., Cleveland, W. G., Jr., Mattingly, G. E., and Hall, J. M., "Air Speed Calibrations at the National Institute of Standards and Technology," *Proceedings of the 1992 Measurement Science Conference* (Anaheim, CA: MSC 1992), pp. 315-326.

2.4.4.3.6 Assuring the quality of test and calibration results

a) The laboratory should have a wind tunnel or similar testing facility. The testing facility should be calibrated using an artifact that is directly traceable to NIST. Calibration of the artifact and the testing facility should be done periodically, with historical data used for process control.

b) The suitable air flow facility should meet certain criteria. The facility should:

1) avoid blocking effects,

2) have good speed control,

3) provide an air stream with spatial uniformity and low turbulence, and

4) monitor environmental conditions.

c) Calibration of the air flow facility should be done periodically, using an artifact calibrated by NIST. The NIST-calibrated artifact should be calibrated periodically.

d) The laboratory should determine the uncertainty of its air speed measurements through artifact calibration by NIST and statistical process control of the testing facility, referenced to accepted methods and practices.

2.4.4.3.7 Accommodation and environmental conditions

a) The laboratory should monitor the environmental conditions of both the measuring area and the interior of the wind tunnel or similar testing device.

b) The laboratory should document and maintain historical reference data regarding the performance of any instrumentation utilized in calibrations of meteorological-type air speed instrumentation.

2.4.4.3.8 Equipment

The laboratory should have the equipment needed to make air speed measurements for the testing facility.

2.4.4.3.9 Test and calibration methods and method validation

a) The laboratory should have a manual outlining the procedures to be followed for each type of calibration.

b) For meteorological-type instrumentation, important considerations would be:

1) procedures for performing the actual calibration,

2) positioning of the instrument under test,

3) determining if the testing facility is calibrated and in control, and

4) fitness of the instrument under test.

c) Other calibration methods, including data reduction and analysis, should be detailed.

d) Calibration methods should be used which identify the performance criteria of the instrument under test that do not, in and of themselves, create situations that affect the performance result. Examples of situations that affect performance are:

1) interference caused by improper placement inside testing facility, or

2) instrument under test unfit for test.

2.4.4.3.10 Handling of calibration instruments

a) Before instruments are calibrated, they should be visually inspected and tested to insure that they are in proper working order.

b) When installing airspeed instrumentation in a testing device, methods should be used which will insure maximum performance of the testing facility and of the instrument under test.

2.4.4.3.11 Reporting the results

a) The reported uncertainty of meteorological-type air speed instrumentation calibrations should be derived from a model of the measurement system which includes, as applicable, uncertainties due to:

1) environmental conditions (i.e., ambient temperature, barometric pressure, and relative humidity, etc.),

2) test conditions (i.e., stagnation temperature, stagnation pressure, relative humidity, etc.),

3) differential pressure of Pitot-static tubes,

4) control of speed of testing device,

5) set-up and actual test,

6) NIST calibration uncertainty, and

7) other factors, as appropriate.

b) Reports and certification should conform to report requirements in ISO/IEC 17025. They should state:

1) laboratory's measurement uncertainty,

2) any applicable traceability information,

3) laboratory calibration method,

4) environmental conditions, and

5) other factors, as appropriate.

2.5 Flow measurement of inert cryogens

2.5.1 Scope

This section sets out the specific technical criteria for a laboratory to be recognized as competent to carry out calibrations of cryogenic flow metering devices. Devices include, but are not necessarily limited to, the following:

a) turbine flowmeters,

b) vortex-shedding flowmeters,

c) ultrasonic flowmeters, and

d) coriolis flowmeters.

2.5.2 Definition

Cryogen: A fluid whose normal boiling point is below 120 K.

2.5.3 Assuring the quality of test and calibration results

2.5.3.1 All sources of variability for the calibration should be monitored (i.e., thermometers, pressure transducers, electronic instrumentation) and check standards should be used to ensure that the calibrations are carried out under controlled conditions. The laboratory should be able to exhibit long term reproducibility of its measurements through statistical control charts. The level of statistical process control should be commensurate with the quoted level of uncertainty for the laboratory's measurements.

2.5.3.2 Whatever methods or artifacts that the laboratory employs for calibrations, these should adequately span the range of the conditions for which the laboratory offers calibrations, and the laboratory should provide an uncertainty value for each of these ranges. The values measured by the methods or artifacts should be recorded and evaluated on an historical basis to determine that the process is in control. These values should be recorded in appropriate formats (for example, graphical plots) to assure that they fall within established control limits.

2.5.4 Accommodation and environmental conditions

2.5.4.1 Special consideration should be given to atmospheric conditions and the local acceleration of gravity if the measurement process is weigh-time based. Due to the nature of cryogenic flows, attention to conditions within the calibration apparatus is a critical design concern, and these conditions (vacuum-jacketed areas) should be controlled and reliably monitored.

2.5.4.2 The laboratory should be able to demonstrate the operation and effectiveness of the vacuum insulation system required in a cryogenic flow measurement system.

2.5.5 Equipment

2.5.5.1 The laboratory should have the equipment needed to make auxiliary measurements (for example, pressure and temperature) of the process fluid.

2.5.5.2 The laboratory should have sufficient equipment to record, evaluate and store properties of the process fluid (for example, the process fluid densities).

2.5.5.3 The laboratory should have the equipment needed to measure atmospheric conditions (for example, temperature, pressure, and humidity).

2.5.5.4 The laboratory should have the equipment needed to measure and record output signals for the range of meters routinely being calibrated.

2.5.5.5 The laboratory flow measurement system should not be operated at flows greater than the specified limit of the system.

2.5.5.6 The laboratory should have a set of procedures necessary for handling and using cryogenic fluids. It should include the following:

a) handling of trapped fluid in fill lines,

b) handling of trapped fluid in the event of power failure,

c) handling of fluid release in the event of vacuum failure,

d) maintaining an adequate ventilation system for fluid released in enclosed environment,

e) maintaining an adequate relief capacity and vent lines,

f) displaying Material Safety Data Sheets for all fluids used in the laboratory,

g) establishing safety procedures in the event of a fluid spill to prevent contact with personnel, and

h) insuring a safe self-regulating storage facility for fluids.

2.5.6 Handling of test and calibration items

Each portion of the measurement system should be used and handled with the proper care to ensure that its performance is not changed. This includes storage for the cryogenic fluid in an environment such that the quality of the material is not degraded.

2.5.7 Personnel

2.5.7.1 Personnel should be trained in handling cryogenic fluids.

2.5.7.2 Personnel should have direct access to Material Safety Data Sheets for all fluids used in the laboratory and should be able to demonstrate knowledge of the information contained therein.

2.5.8 Test and calibration methods and method validation

2.5.8.1 The method for conducting the calibration should be rigorously specified and well documented.

2.5.8.2 Tests should be statistically designed to adequately quantify all variables in the calibration.

2.5.8.3 Calibration tests should be designed to satisfactorily reproduce results for flow rate, temperature, and pressure.

2.5.8.4 The laboratory should be able to vary the flow, temperature, pressure, and subcooling, and also demonstrate long-term stability in these quantities.

2.5.8.5 The laboratory should be able to demonstrate how it checks for, characterizes, and accounts for heat leaks in the meters calibrated.

2.5.9 Records

2.5.9.1 The laboratory should measure and record all appropriate data and information during the calibration procedure so that results can be reevaluated if a facility problem is found after final results are produced.

2.5.9.2 All piping configurations used during a calibration should be specified in order to account for possible differences found between the calibration results and the field records for the meters tested.

2.5.9.3 Records associated with any standards used in the laboratory should be maintained in appropriate formats for the period of time specified in the laboratory's quality manual.

2.5.10 Reporting the results

2.5.10.1 The laboratory should provide an uncertainty statement under the guidelines set forth by NIST Technical Note 1297. The uncertainty should include contributions due to:

a) master artifact,

b) all property measurement devices,

c) all electronic instrumentation,

d) fluid property standards,

e) all dimensional measurements, and

f) the uncertainty contributed by NIST's calibration.

2.5.10.2 Laboratories should maintain calibration certificates on all associated instruments used for its flow measurement capability, as well as for data acquisition.

2.5.10.3 The laboratories should document calibrations for all associated instrumentation that contributes to the uncertainty of its flow measurement capabilities.

2.6 Flow calibrations

2.6.1 Scope

2.6.1.1 This section sets out the specific technical criteria for a laboratory to be judged competent to carry out calibrations of flow meters. Accreditation is offered for laboratories calibrating the following types of flow meters:

a) differential pressure producers,

b) turbine meters,

c) variable area meters,

d) sonic nozzles,

e) ultrasonic flow meters,

f) positive displacement meters,

g) vortex-shedding devices,

h) magnetic flow meters,

i) coriolis meters,

j) thermal meters, and

k) target meters.

2.6.1.2 The artifact calibration facilities currently included in the accreditation program are:

a) Small Air Flow Facilities - for flow rates up to 2.8 standard cubic meters per minute (100 SCFM),

b) Large Air Flow Facilities - for flow rates from 0.99 to 84.95 standard cubic meters per minute (35 to 3000 SCFM) at pressures up to 620 KPa (90 psig),

c) Water Flow Facilities - for pipeline diameters up to 0.4 meters (16 inches), and

d) Hydrocarbon Liquid Flow Facilities - both volumetric and gravimetric systems using JP-4 and JP-5 jet fuels or their equivalents. The flow rate range is 3.5×10^{-5} to 1.5 cubic meters per minute (0.01 to 400 gpm).

2.6.2 References

2.6.2.1 General references

a) NIST SP 250, "NIST Calibration Services Users Guide," available on line at http://ts.nist.gov/ts/htdocs/230/233/calibrations/.

b) Mease, N. E., Cleveland, Jr., W. G., Mattingly, G. E., and Hall, J. M., "Air Speed Calibrations at the National Institute of Standards and Technology," *Proceedings of the 1992 Measurement Science Conference*, Anaheim, CA, 1992.

c) Goldstein, Richard J., editor, *Fluid Mechanics Measurements*, Hemisphere Publ. Co., New York, NY, 1979 (see Chapter 6 on Volume Flow Measurements).

d) Spitzer, D. W., editor, *Flow Measurement - Practical Guides for Measurement and Control*, Instrumentation Society of America, Research Triangle Park, NC, 1991 (see Chapter 24).

e) DeCarlo, Joseph P., "Fundamentals of Flow Measurement," Instrumentation Society of America, Research Triangle Park, NC, 1984.

f) Miller, R. W., *Flow Measurement Engineering Handbook*, McGraw Hill, 1983.

2.6.2.2 Gas flow calibration references

a) Johnson, A. N., Wright, J. D., Moldover, M. R., and Espina, P. I., "Temperature Characterization in the Collection Tank of the NIST 26 m^3 PVTt Gas Flow Standard," *Metrologia,* **40** (2003), pp. 211-216.

b) Wright, J. D., "What is the 'Best' Transfer Standard for Gas Flow?," *Proceedings of the 2003 FLOMEKO* (Groningen, Netherlands: Gasunie 2003).

c) Wright, J. D., Johnson, A. N., and Moldover, M. R., "Design and Uncertainty Analysis for a PVTt Gas Flow Standard," *NIST J. of Res.,* **108**, *1* (2003), pp. 21-47.

d) Wright, J. D., and Espina, P. I., "Flow Calibration Services at NIST," *Proceedings of the ISA 46th International Instrumentation Symposium* (Seattle, WA: ISA 2000).

e) Wright, J. D., and Johnson, A. N., "Uncertainty in Primary Gas Flow Standards Due to Flow Work Phenomena," *Proceedings of FLOMEKO 2000* (Salvador, Brazil: IPT 2000).

f) Nakao, S., Wright,J. D., Barbe, J., Niederhauser, B., Quintilii, M., and Knopf, D., "Intercomparison Tests of the NRLM Transfer Standard with the Primary Standards of NIST, BNM-LNE, OFMET and PTB for Small Mass Flow Rates of Nitrogen Gas," *Proceedings of the Metrologie '99 Conference* (Paris, France: 1999).

g) Wright, J. D., Mattingly, G. E., Nakao, S., Yokoi, Y., and Takamoto, M., "International Comparison of a NIST Primary Standard with an NRLM Transfer Standard for Small Mass Flow Rates of Nitrogen Gas," *Metrologia*, **35** (1998), pp. 211-221.

h) Wright, J. D., and Mattingly, G. E., "NIST Calibration Services for Gas Flow Meters: Piston Prover andBell Prover Gas Flow Facilities," *NIST SP 250-49* (Gaithersburg, MD: NIST 1998).

2.6.2.3 Liquid flow calibration references

a) Gowda, V., Yeh, T. T., Espina, P. I., and Yende, N. P., "The New NIST Water Flow Calibration Facility," Proceedings of the 2003 FLOMEKO (Groningen, Netherlands: Gasunie 2003).

b) Yeh, T. T., Yende, N. P., and Espina, P. I., "Theoretical Self-Error-Canceling Diverters for Liquid Flow Calibration Facilities," *Proceedings of the 2003 FLOMEKO* (Groningen, Netherlands: Gasunie 2003).

c) Yeh, T. T., Yende, N. P., Johnson, A., and Espina, P. I., "Error Free Liquid Flow Diverters for Calibration Facilities," *Proceedings of the 2002 ASME Fluids Engineering Division Summer Meeting* (Montreal, Quebec, Canada: ASME 2002), FEDSM2002-31085.

d) Wright, J. D., and Espina, P. I., "Flow Calibration Services at NIST" *Proceedings of the ISA 46th International Instrumentation Symposium* (Seattle, WA: ISA 2000).

e) Mattingly, G. E., "The Characterization of a Piston Displacement-Type Flowmeter Calibration Facility and the Calibration and Use of Pulsed Output Type Flowmeters," NIST J. of Res., **97**, *5* (1992), pp. 509-531.

2.6.3 Assuring the quality of test and calibration results

2.6.3.1 All sources of measurement variability associated with the calibration should be monitored (e.g., thermometry, pressure-measuring instrumentation, humidity-measuring devices, etc.), and check standards should be used to ensure that the calibrations are carried out under controlled conditions. The laboratory should maintain statistical process control (SPC) commensurate with the uncertainty levels needed for the calibration. The SPC control parameters should be based on measurements of check standards (or closure parameters); the imprecision of these parameters can be quantified using multiple measurements. Type B uncertainties of the measurement process should be quantified using the true value of the result or an adequate estimate of it. The frequency and number of process control checks should be appropriate for the level of uncertainty and reliability claimed for the calibration.

2.6.3.2 The laboratory should have control artifacts which adequately span the range of materials and conditions normally calibrated by the laboratory. Every measured value of each variable should be recorded and compared to past values to determine whether or not the process is in control. These current and past values should be plotted on a control chart (may be done on a computer and stored electronically) that has upper and lower control limits.

2.6.3.3 The laboratory should maintain control charts on all of the calibration equipment. These should show traceability to measurement values obtained from NIST or other national measurement institute to establish and demonstrate appropriate statistical process control. The uncertainty of all of the components of the measurement process should be quantified using multiple measurements. Type B uncertainties of the measurement process should be quantified using the true value of the result or an adequate estimate of it.

2.6.4 Accommodation and environmental conditions

2.6.4.1 The environmental conditions (i.e., temperatures, pressures, relative humidity, etc.) in the flow meter calibration area should be controlled and reliably measured, with maximum permitted variations depending on the conditions of and the uncertainty level needed for the calibration.

2.6.4.2 The laboratory should be adequately sized and arranged to accommodate all types and sizes of flowmeter assemblies that it plans to calibrate. The temperature should be sufficiently stable and near appropriate reference temperatures chosen for reported results.

2.6.5 Equipment

2.6.5.1 The laboratory should have the equipment needed to make auxiliary measurements on pertinent metering assemblies or artifacts (e.g., flow measuring facility, and the measurement techniques and associated instrumentation needed for such a testing facility).

2.6.5.2 The laboratory should have atmospheric condition measuring capabilities suitable to the calibration procedure (e.g., for barometric pressure measurement, temperature measurement, relative humidity measurement, etc.).

2.6.5.3 A laboratory that certifies artifacts to tolerances should demonstrate a measurement uncertainty which does not exceed 50% of the tolerance. Exceptions to this ratio will be accepted for measurement systems which are documented to be state-of-the-art.

2.6.5.4 The laboratory should have all the equipment necessary to make all of the parameter measurements needed for the meter under test, as well as the flow standard-method equipment available.

2.6.5.5 The laboratory should have the capability to determine the quality of its flow measurements through periodic artifact calibration by NIST or other national measurement institute. Statistical process control of the testing facility should be referenced to accepted methods and practices. These should be based upon appropriate conservation of mass principles.

2.6.6 Test and calibration methods and method validation

2.6.6.1 The laboratory should have a manual outlining the procedures to be followed for each type of calibration.

2.6.6.2 Calibration methods should identify the performance criteria of the laboratory standard vs. the meter under test. These methods should not, in and of themselves, create situations which affect the performance result.

2.6.7 Handling of test and calibration items

2.6.7.1 Flow metering artifacts should be cleaned and stored in a manner to prevent accidental contact with materials which could cause damage to them.

2.6.7.2 Flow metering artifacts should not be used beyond their limits.

2.6.7.3 Flow metering artifacts should be inspected for deficiencies which could otherwise damage either the performance of the artifact or the testing facility.

2.6.7.4 Before items are calibrated, they should be visually inspected and tested to insure that they are in proper working order.

2.6.7.5 When installing flow meters in test facilities, methods should be used which are appropriate for the conditions planned for the use of the meter, and which will insure satisfactory performance of the testing facility and the meter.

2.6.8 Test and calibration methods and method validation

The reported uncertainty should be derived from a complete model of the measurement system that includes, as applicable, the uncertainties due to:

a) master artifact,

b) long-term reproducibility of measurement system,

c) statistics for multiple measurements at each flow rate,

d) environmental conditions,

e) inlet pipe profile,

f) flow pulsation effects,

g) pipe vibration effects, and

h) unsteadiness in pipe flow.

2.6.9 Reporting the results

2.6.9.1 The reported uncertainty of flow calibrations should be derived from a complete model of the measurement system which includes, as applicable, uncertainties due to:

a) environmental conditions (i.e., ambient temperature, barometric pressure, relative humidity, etc.),

b) appropriate test conditions (i.e., temperatures, pressures, relative humidity, etc.),

c) performance of the meter under tests,

d) connecting pipework effects,

e) uncertainty of NIST or other national measurement institute calibration,

f) flow profile in test piping,

g) personnel effects, and

h) software verification.

2.6.9.2 In addition to the report requirements of ISO/IEC 17025, the calibration reports should provide:

a) testing facility information such as the fluid and flow conditions,

b) all applicable traceability information,

c) performance of the meter under test,

d) environmental conditions,

e) inlet pipe profile,

f) flow pulsation effects,

g) pipe vibration effects, and

h) unsteadiness in pipe flow.

2.7 Force calibrations

2.7.1 Scope

The purpose of this section is to specify the technical criteria needed to meaningfully assess the competence of a calibration laboratory that performs calibrations of force measurement systems.

2.7.2 References

a) ASTM E 74-00, *Standard Practice of Calibration of Force Measuring Instruments for Verifying the Force Indication of Testing Machines.*

b) A New Statistical Model for the Calibration of Force Sensors, *NBS Technical Note 1246*, NBS, June 1988.

c) Mass Calibrations, *NIST Special Publication 250-31*, NIST (formerly NBS), January 1989.

d) ASTM E 617-97, *Standard Specification for Laboratory Weights and Precision Mass Standards.*

e) ASTM E 4-99, *Standard Practices for Load Verification of Testing Machines.*

2.7.3 Accommodation and environmental conditions

2.7.3.1 All instruments should be allowed sufficient time to reach room temperature prior to being calibrated. The recommended room temperature is 23 ± 2.0 °C (73.4 °F).

2.7.3.2 During calibration the temperature should be monitored close to the instrument. A temperature stability of ± 0.5 °C should be maintained. However, if the temperature variations exceed ± 0.2 °C during the calibrations of non-temperature compensated instruments, such as proving rings, the calibration data should be corrected in accordance with section 9 of ASTM E 74-95.

2.7.4 Equipment

2.7.4.1 Primary force standard definition

In accordance with ASTM E 74-00, a primary force standard is: "a deadweight force applied directly without intervening mechanisms such as levers, hydraulic multipliers, or the like, whose mass has been determined by comparison with reference standards traceable to national standards of mass." Accordingly, laboratories that perform primary force standards calibrations should do so with deadweights that apply the force directly. Primary force standard deadweight machines cannot have any mechanism for amplifying the force such as levers, hydraulic multipliers or the like, or any mechanism that counterbalances the frame (or tare).

2.7.4.2 Primary standard weight construction

Weights used as primary standards in deadweight machines should be made of rolled, forged, or cast metal. The surface roughness of the weights should not exceed 63 microinches. If the weights are plated, painted or coated, the finish should be of a proven design and material. Acceptable materials include cadmium, nickel-chromium or other stable metallic plating and aluminum/varnish or other metallic paints. The forces developed by the weights should be determined using the formula given in ASTM E 74-00, section 6.1. This implies that the laboratory should have knowledge of the local gravity and its uncertainty.

2.7.4.3 Primary standard weight uncertainty

The masses of the weights should be known to within 0.005 % of their nominal values by comparison to reference standards traceable to NIST or equivalent national measurement laboratory. The laboratory should keep records of the calibration of all weights. The uncertainty of the vertical component of force applied by each weight should also be stated in the laboratory records and in reports of tests.

2.7.4.4 Primary standard calibration intervals

The masses of the weights should be determined every 5 years except for deadweight machines whose weights are made up of high grade stainless steel that are not coated such as grade AISI 303, 304 and 410. For large dead weight machines, where disassembly is not practical, it is acceptable to verify the primary standard through comparison calibrations with NIST (or equivalent) using ASTM E 74-00 as a guide. The uncertainty of the comparison should not exceed 50.0×10^{-6} for a primary force standard.

2.7.4.5 Secondary standard definition

In accordance with ASTM E 74-00, a secondary force standard is: "An instrument or mechanism, the calibration of which has been established by comparison with primary force standards." Laboratories that calibrate other force measuring instruments should use either primary standards or secondary standards as defined above. Secondary standards having capacities up to 1,000,000 lbf (4.4MN) should be calibrated by primary standards (by deadweights) whose masses have been determined in accordance with section 2.7.4.4. Secondary force standards can only be used with a suitable loading frame or mechanism to insure a safe axial force generation to the system under test. The system should exhibit no parasitic, frictional or mechanical losses during use. The loading system should be supplied with well marked accessory fittings to enable proper and safe calibrations to be performed.

2.7.4.6 Secondary standard uncertainty

The uncertainty of instruments used as secondary standards should not exceed 0.05 % of the applied force. Moreover, in accordance with the requirements set forth in ASTM E 74-00, section 8.5.2.1, the lower limit of the secondary standard should be 2000 times the uncertainty obtained from its calibration data.

2.7.4.7 Secondary standard calibration intervals

The time interval between calibrations of all secondary standards should not exceed 24 months. The laboratory should keep records of the secondary standards calibrations.

2.7.4.8 Instruments used to verify testing machines, uncertainty

Instruments used to verify testing machines in accordance with ASTM E 4-97 may be calibrated either by deadweights or with secondary standards. The uncertainty of the instrument as determined from the calibration data should not exceed 0.25 % of force. In accordance with ASTM E 74-00 section 8.5.2.2, the lower limit of the instrument should be 400 times the uncertainty obtained from its calibration data.

2.7.4.9 Instruments to verify testing machines, calibration intervals

The time interval between calibrations of these instruments should not exceed 2 years provided that the changes between calibration results do not exceed 0.1 % of capacity. When this criteria is exceeded the frequency of calibration should not exceed 12 months. The laboratory should keep all records of calibration for inspection.

2.7.4.10 Overloaded or repaired instruments

Any force standard or multiplying system that is repaired or modified in a way that may result in changes in the calibration curve should be recalibrated prior to use. Any instrument that sustains an overload that produces a change in the zero load output of 1 % or more should be recalibrated prior to use.

2.7.4.11 Accessory hardware

All calibration hardware that is subject to calibration forces such as coupling nuts, pullrods, adapters, etc., should be clearly labelled with the maximum allowable load they can sustain.

2.7.4.12 Electrical instruments calibration

The time interval between calibrations of the electrical instrumentation used to calibrate load cells in voltage ratio mode should not exceed 12 months. The laboratory should keep records of calibration. Moreover, the laboratory should demonstrate that it can monitor the instrument stability between the calibration intervals.

2.7.4.13 Electrical instruments uncertainty

The uncertainty of the electrical instrumentation should not exceed 0.10 μV/V.

2.7.5 Test and calibration methods and method validation

2.7.5.1 Preferred method

The preferred calibration method is that described in ASTM E 74-00; however, other methods are allowed. All laboratories should have a written description of the procedures followed for each type of calibration.

2.7.5.2 Distribution and number of calibration forces

The calibration forces should be distributed over the full range of the instrument, preferably at 10 percent intervals throughout the range. Limitations in equipment may prohibit equal spacing of the forces; accordingly, equal spacing, while desirable, is not required. A minimum of at least 30 applications of force should be applied; of these, a minimum of 10 should be at different forces. Each calibration sequence should be applied at least twice in order to quantify the instrument reproducibility in the replicated conditions.

2.7.5.3 Randomization of loading conditions

Randomization of loading conditions is of primary importance. The instrument undergoing calibration should be rotated in the calibration machine in accordance with the recommendation contained in ASTM E 74-00 section 7.5. In calibrations involving both compression and tension modes, part of the compression calibration should be conducted before the tension calibration and part after, in order to quantify any hysteresis effects that may affect the instrument.

2.7.6 Records

All measurements should be appropriately recorded. The data sheets or the computer file containing the data should include the serial number of the device, the temperature during the calibration, the date of calibration and other pertinent information. Records of calibrations should be maintained for a minimum of 5 years. Records associated with secondary standards should be kept for the entire lifetime of that standard.

2.7.7 Reporting the results

In addition to the report requirements of ISO/IEC 17025, the laboratory should provide calibration reports that conform to the guidelines of ASTM E 74-00. The calibration report should also contain the following information:

a) manufacturer and serial number of the instrument calibrated,

b) type of reference standard used (i.e., primary standard, secondary standard), including the uncertainty in the applied forces,

c) temperature at which the calibration was performed, including limits of temperatures variations during the calibrations,

d) listing of the calibration forces applied and deflections observed,

e) the calibration curve, including the method of analysis used to obtain the curve, and the deviations of the experimental data for the fitted curve, and

f) the uncertainty associated with the calibration results and limits of class A and AA loading ranges if such limits are required.

2.8 Mass calibrations

2.8.1 Scope

The purpose of this section is to specify the specific technical criteria needed to meaningfully assess the competence of a calibration laboratory that performs mass calibrations.

2.8.2 Background

2.8.2.1 A laboratory should declare its measurement capability in terms of uncertainties for each mass value. For many laboratories these uncertainties correspond to three arbitrary echelons of measurements that roughly correspond to weight classifications at nominal mass value ranges of measurements. For laboratories seeking accreditation at an uncertainty range that corresponds to a specific echelon, the scope of accreditation should follow the echelons as defined in Table 1 with a declared range of nominal mass values and their associated uncertainties. Recommended ranges of mass values are provided in Table 2. A summary of section 2.8.4, 2.8.6.1, 2.8.6.6, and 2.8.7.1 is provided in Table 5.

2.8.2.2 The echelon stated by the laboratory is associated with the standards, procedures, measurement control, facilities, equipment, staff capability, and the overall level of performance described in Table 1, and is specifically defined by the expanded uncertainty. Echelons are evaluated by all of these factors in addition to the laboratory's reported uncertainties.

Table 1. Mass calibration echelons

Echelon	Typical Test & Verification Levels	Expanded Uncertainty of the Measurement Results
I, (Extra Fine)	OIML, Classes E_1, E_2 ASTM, Classes 0, 1	The expanded uncertainty must be less than 1/3 of stated tolerances at all levels.
II, (Fine)	OIML, Classes F_1, F_2 ASTM, Classes 2, 3	
III, (Medium)	OIML, Classes M_1,M_2,M_3 ASTM, Classes 4, 5, 6, 7 NIST, Class F	

Table 2. Typical scopes of accreditation for mass calibrations

Echelon	Scope of Accreditation Nominal Value Ranges	
I	≥ 30 kg (define limit)	
	30 kg to 1 mg	
	1 kg to 1 mg	
	100 g to 1 mg	
II	≥ 30 kg (define limit)	For special applications:
	30 kg to 1 mg	≥ 1000 lb (define limit)
	1 kg to 1 mg	≥ 50 lb (define limit)
	100 g to 1 mg	50 lb to 0.001 lb
III	≥ 30 kg (define limit)	For normal applications:
	30 kg to 1 g	≥ 1000 lb (define limit)
	1 kg to 1 g	≥ 50 lb (define limit)
	100 g to 1 g	50 lb to 0.001 lb

2.8.2.3 The reported uncertainty of mass standards calibrated by a mass calibration laboratory will vary depending on available balances, the uncertainty of reference standards, and the nominal value of the mass standard being tested. Thus, a laboratory may perform calibrations of Echelon I in some ranges, for example

at 1 kg, and may perform calibrations at Echelon II, e.g., 20 kg, in other ranges. The laboratory performs calibrations in a specified range as requested; however, all laboratories may not be capable of meeting the requirements of all echelons. Differing equipment, skills, knowledge, measurement control, and demonstrated competence are required for each of the echelons. Demonstrated competence in one echelon is insufficient to guarantee adequate performance in the others.

2.8.3 References

a) ANSI/ASTM E 617-97, *Standard Specification for Laboratory Weights and Precision Mass Standards*, 1997.

b) International Organization for Legal Metrology (OIML) IR 111, *Weights of Classes E_1, E_2, F_1, F_2, M_1, M_2, M_3*, 1994 [under revision in 2003].

c) Harris, G. L., editor, NIST Handbook 105-1, *Specifications and Tolerances for Reference Standards and Field Standard Weights and Measures, Specifications and Tolerances for Field Standard Weights, (NIST Class F)*, 1990.

d) Harris, G. L., Torres, J. A., "Selected Laboratory and Measurement Practices, and Procedures, to Support Basic Mass Calibrations," *NISTIR 6969*, 2003.

e) Cameron, J. M., Hailes, G. E., Designs for the Calibration of Small Groups of Standards in the Presence of Drift, *NBS Technical Note 844*, 1974.

f) Cameron, J. M., Croarkin, M. C., Raybold, R. C., Designs for the Calibration of Standards of Mass, *NBS Technical Note 952*, 1977.

g) Fraley, K. L., Harris, G. L., "Advanced Mass Calibrations and Measurement Assurance Program Requirements for State Calibration Laboratories," *NIST IR 5672*, 1995.

2.8.4 Assuring the quality of test and calibration results

2.8.4.1 Appropriate measurement control programs should be in place and available for review for each echelon and nominal mass range for which calibration data is provided. Note Table 5 for appropriate measurement control programs for each echelon. Appropriate data include balance standard deviations that represent process variation and well-characterized check standard values.

2.8.4.2 Measurement control techniques should exhibit results consistent with the procedures used to perform calibrations and should be integrated into the measurement procedures to accurately reflect the measurement process. For those situations where statistical information is not inherent to the process, i.e., simple measurements without built-in redundancy checks, additional measurements should be made to provide experimental characterization of the measurement sufficient for an adequate estimation of the process uncertainty. Those data should be available for review.

2.8.5 Accommodation and environmental conditions

2.8.5.1 To be deemed capable of making adequate measurements, calibration laboratories should provide an environment with adequate environmental controls appropriate for the level of measurements to be made, according to echelon classes defined herein. The environmental conditions are summarized in Table 3.

Table 3. Summary of environmental parameters

Echelon	Temperature	Relative Humidity
I	20 to 23 °C, a set point ± 1 °C, maximum rate of change 0.5 °C/h	40 % to 60 % ± 5 % per 4 h
II	20 to 23 °C, a set point ± 2 °C, maximum rate of change 1.0 °C/h	40 % to 60 % ± 10 % per 4 h
III	18 to 27 °C, maximum rate of change 2 °C/h	40 % to 60 % ± 20 % per 4 h

NOTE The environmental conditions should also be within the specifications of the weighing instruments where applicable.

2.8.5.2 Cleanliness guidelines are usually met without clean-room type air handling systems by maintaining clean-room type practices. Excessive air exchange rates negatively affect balance performance. The laboratory should maintain limited access to the calibration area and minimize contamination (provide a clean surface) for locations where calibration items are being tested. Activities such as smoking, eating, or drinking and items such as paper products, printers and files contribute to the difficulty of maintaining adequate cleanliness and are not recommended. A positive pressure, laminar-type air flow is usually needed to maintain cleanliness recommendations and to minimize air currents.

2.8.5.3 Vibration should not diminish the performance of precision analytical balances and mass comparators. Proximity to heavy machinery, railways, heavily travelled highways, or similar sources of known vibration is not recommended. Steps are often taken to attenuate vibration to an acceptable level of stability with methods such as massive piers (solid marble or concrete tables), isolated foundations, or elimination of the vibration source. Balances and mass comparators used for Echelons I and II generally require massive piers, independent piers, and/or an isolated foundation; pneumatic or hydraulic tables are inappropriate.

2.8.5.4 Undesirable effects due to static electricity should be controlled, if needed, with methods such as humidity, anti-static deionizing radiation devices, the grounding of balances or operators, or with the use of special conductive flooring, and selection of proper clothing for staff.

2.8.6 Equipment

2.8.6.1 Minimum reference standards should be available at each echelon and range for which the laboratory is accredited, as recommended in Table 5. Sufficient historical data and uncertainty analysis should be available to support the standards used.

2.8.6.2 Due to algorithms used in mass calibration, the uncertainty of measurement results from auxiliary instruments for Echelons I and II, (e.g., scale, analytical balance, mass comparator) is less important than the precision of the instrument. However if such equipment is repaired, it should be reevaluated to ascertain its current level of precision prior to use, and the uncertainty estimate should reflect the post-repair performance.

2.8.6.3 The precision of the scale, analytical balance, or mass comparator, as determined through appropriate process control charts, should be suitable to the echelon class for which it is used. For an

application where external standards are used for comparison, appropriate control charts should be maintained to evaluate the process standard deviation. Note Table 5 requirements for further evaluation.

2.8.6.4 Means should be provided to measure barometric air pressure, air temperature, and relative humidity of the laboratory environment as indicated in Table 4; documentation of the uncertainty and traceability of these measurement results is required. These instruments should be used in close proximity to the balance being used. For Echelon I, temperature may be measured inside the weighing chamber when there is a difference between the air temperature in the balance chamber and the surrounding area. For Echelon III, where buoyancy corrections are generally negligible, recording environmental data is useful in the support of general environmental requirements of the previous section, but the accuracy of these data generally does not affect measurement results.

Table 4. Summary of accuracy requirements for secondary auxiliary equipment

Echelon Parameter	Barometric Pressure	Temperature	Relative Humidity
I	± 65 Pa (± 0.5 mm Hg)	± 0.1 °C	± 5%
II	± 135 Pa (± 1.0 mm Hg)	± 0.5 °C	± 10%
III	The laboratory maintains documented measurement results uncertainty.		

2.8.6.5 For Echelon I, the laboratory should state the presence of a possible systematic error in the combined uncertainty associated with the use of an assumed density in the primary or reference standards (additional type B component) or the laboratory should have appropriate means to measure the density of mass standards. If the magnetic susceptibility of the mass standards is evaluated, it should be indicated on calibration reports. The methods used to determine density or magnetic susceptibility should be documented.

2.8.6.6 Each mass standard used as a reference standard by the laboratory should be calibrated by NIST or by an accredited laboratory with capability adequate to sustain the uncertainty required and maintain traceability to BIPM. The laboratory should provide evidence, such as periodic surveillance, that the standard is, in principle, acceptable for providing calibration services at each echelon. Note Table 5 for traceability requirements.

2.8.6.7 Balances used as a direct comparison to the mass unit should be given a verification test or calibration prior to use. For an application requiring balance accuracy, the laboratory should choose appropriate and correct calibration algorithms. Balances used as dividers and multipliers of the mass unit should be capable of providing the appropriate uncertainty and linearity requirements of the echelon class for which they are used. Calibration of built-in standards should be performed periodically and should be verified prior to use. History from measurement control programs (surveillance testing) may be used to determine calibration intervals.

2.8.6.8 Measurement results data from instruments used to monitor environmental conditions in the laboratory should be traceable to a suitable national laboratory (directly or by way of an accredited laboratory) and these instruments should be recalibrated periodically unless intrinsic (defining) standards are employed. Calibration and intercomparison periods should be documented by the laboratory. For intrinsic standards, data should be available of intercomparison with standards of known measurement values.

2.8.7 Test and calibration methods and method validation

2.8.7.1 The algorithm chosen for the mass measurement, the reference standard(s) to be used, and the equipment to be used for a particular calibration should provide acceptable levels of uncertainty for that calibration. A documented procedure should be available in the laboratory to determine the correct algorithm. Note Table 5 for guidelines.

2.8.7.2 Computer programs should have passed software quality analysis. Computer programs should be tested, using standard data sets designed to magnify errors, as an effective way of showing that program errors are not present which do not affect some measurements but cause others to be incorrect. Computer programs should be documented in detail. The documentation should include technical references that provide the basis for the algorithm, the weighing equation, and the data set used to test the program for errors.

2.8.8 Handling of test and calibration items

2.8.8.1 The laboratory should have documented procedures to ensure adequate chain-of-custody of calibration items if required by law.

2.8.8.2 The laboratory will document appropriate procedures to ensure that cleaning, if performed, ensures the integrity of the standards, and to provide for thermal conditioning, where appropriate. The laboratory must allow adequate stabilization time for mass standards to ensure environmental and thermal stability prior to calibration.

2.8.8.3 Documented procedures to ensure adequate tracking of calibration items should be appropriate to the class of mass standard. Strings, tags, or labels fastened to the standard are inappropriate.

2.8.9 Reporting the results

2.8.9.1 Certificates and reports should describe the mass standards mentioned in the report with sufficient detail to avoid any ambiguity. In addition to the general report requirements of ISO/IEC 17025, for Echelon I and II calibration, additional items to be included on a test report, are:

a) mass (true mass) values,

b) conventional (apparent) mass values versus appropriate reference density,

c) reference density,

d) uncertainties,

e) material,

f) thermal coefficient of expansion (if used in calculations),

g) construction,

h) density (measured or assumed),

i) any identifying markings, and

j) tolerances, if appropriate.

2.8.9.2 Environmental parameters measured during the test should be provided on certificates and reports for Echelons I and II. Typical ranges are acceptable for Echelon III. These include:

a) laboratory temperature,

b) barometric pressure, and

c) relative humidity.

2.8.9.3 Information regarding cleaning methods should be provided on the test report.

2.8.9.4 Reports may include reference to OIML or ASTM classification schemes and tolerances. Items being calibrated should meet appropriate specifications for evaluation as well as tolerances. It is the responsibility of the requestor of the calibration to select classifications acceptable for their needs.In the instances where magnetism, surface finish, and density are not tested for Echelon I and II, a statement to that effect should be included on the calibration report.

2.8.9.5 The external surface of a mass standard should be free of any sign of abuse or damage. Signs of abuse or misuse include the placement of labels, tags, wires or other material on mass standards. In addition, visible dirt and fingerprints are a sign of misuse for Echelons I and II. It is recommended that the calibration laboratory establish appropriate means for notifying customers regarding any unusual factors, such as signs of abuse regarding the mass standard being tested, and this should be included on the report. Out-of-tolerance conditions should be reported, as well.

Table 5. Measurement control, standards, traceability, calibration methods

Echelon	Minimum Measurement Control	Minimum Reference Standards	Minimum Traceability	Minimum Calibration Methods
I (Extra Fine)	Process control charts Check standards for each decade, with long term standard deviation Surveillance of all standards Proficiency testing On-site assessment Participation in interlaboratory comparisons	OIML Class E_1, or E_2 or ASTM Class 0, 1 or Single piece, highly polished	NIST, or other national level calibration every 2-5 years based on measurement process data and independent verification, or NIST Mass MAP, or User-operated mass calibration package, on-site to verify traceability (when available).	Documented weighing designs consisting of redundant comparisons, with built-in process controls such as those used at the national level. For example: Technical Notes, 952 and 844, SOP 28
II (Fine)	Process control charts Check standards for each decade, with long term standard deviation Surveillance of selected standards Proficiency testing On-site assessment Participation in interlaboratory comparisons	OIML Class E_1, or E_2 ASTM Class 0, 1, or Single piece, highly polished	NIST calibration every 2 - 5 years based on measurement process data and independent verification, or Calibration by accredited Echelon I laboratory, if uncertainty requirements can be met.	Documented comparison calibration procedure For example: NBS Handbook 145, double substitution, or 3-1 weighing design, or equivalent SOP 4, SOP 5
III (Medium)	Process control charts Check standards for each balance Proficiency testing On-site assessment Participation in interlaboratory comparisons	Working standards such as: ASTM Class 2, 3 or OIML Class F_1 Two piece acceptable, fine finish	Calibration of all working standards by NIST every 2 - 5 years based on measurement process data and independent verification, or Calibration of all working standards by an Echelon I or II accredited or recognized labs every 2 - 5 years based on measurement process data and independent verification	Use of annually calibrated balance with documented verification procedure prior to each use Use of modified substitution (NIST Handbook 145) Other documented and verified procedure

2.9 Volume calibrations

2.9.1 Scope

2.9.1.1 The purpose of this section is to provide the specific technical criteria needed to meaningfully assess the competence of a calibration laboratory that performs volume calibrations. It should be noted that the type of calibration procedure affects the achievable uncertainty.

2.9.1.2 Volumetric measurements are obtained from mass measurements of known-density materials. Volume calibrations may be determined by either a gravimetric (weighing procedure) or a volume transfer (comparative) method. The two methods have different technical requirements and both are defined here.

2.9.1.3 The measurement of volume by flow metering methods (and flow meter calibration) is outside the scope of this section.

Table 1. Recommended scope of accreditation for given type of calibration procedure

Type of Procedure	Scope of Accreditation - Nominal Value Range		
Gravimetric	Glassware		Metal Test Measures or Provers
	Standard	Pipets & Syringes	
	≥ 1 L		≥ 2000 L **or** ≥ 500 gal
	1 L to 100 mL		100 L < V < 2000 L **or** 25 < V < 500 gal
	100 mL to 1 µL	100 mL to 1 µL	≤ 100 L **or** ≤ 25 gal
Volume Transfer	Glassware		Metal Test Measures or Provers
	≥ 1 L **or** 1 qt		≥ 2000 L **or** ≥ 500 gal
	100 mL < V < 1 L **or** 1 gill < V < 1 qt		100 L < V < 2000 L **or** 25 < V < 500 gal
	...		≤ 100 L **or** ≤ 25 gal

2.9.2 Background

2.9.2.1 The gravimetric procedure is based on the conservation of mass principle where a determination of the mass of water contained in or delivered from the vessel that is being calibrated is used to define volume. The mass values are determined in air, are corrected for air buoyancy effects, and are corrected to appropriate reference temperatures. The uncertainty for the measurement results will vary depending on balances used, the purity of the water, the ability to make accurate temperature measurements, the nominal value of the volume standard being tested, and the ability to make adequate mass measurements.

2.9.2.2 In the volume transfer procedure, water is delivered from a reference volume standard to the vessel under test. Temperature corrections are made to compensate for the cubical coefficients of thermal expansion of the standard, test vessel, and water to a specified reference temperature. The uncertainty of the measurement results will vary considerably depending on the presence of a meniscus, the cleanliness and

drain characteristics of the container, the cleanliness and purity of the water, and the ability to make adequate temperature measurements.

2.9.3 References

a) ANSI/ASTM E 287-02, *Standard Specification for Laboratory Glass Graduated Burets.*

b) ANSI/ASTM E 288-94 (1998), *Standard Specification for Laboratory Volumetric Glass Flasks.*

c) ANSI/ASTM E 438-92 (2001) e1, *Standard Specification for Glasses in Laboratory Apparatus.*

d) ANSI/ASTM 542-01, *Standard Practice for Calibration of Laboratory Volumetric Apparatus.*

e) ANSI/ASTM 694-99, *Standard Specification for Laboratory Glass Volumetric Apparatus.*

f) ANSI/ASTM 969-02, *Standard Specification for Glass Volumetric (Transfer) Pipets.*

g) OIML IR 4, *Volumetric flasks (one mark) in glass*, 1972.

h) OIML IR 40, *Graduated Pipettes for Verification Officers,* 1982.

i) OIML IR 41, *Standard Burettes for Verification Officers*, 1981.

j) OIML IR 43, *Standard Graduated Glass Flasks for Verification Officers*, 1981.

k) Harris, G. L., editor, *NIST Handbooks 105-2, 105-3, 105-4*, "Specifications and Tolerances for Reference Standards and Field Standard Weights and Measures: Specifications and Tolerances for Field Standard Measuring Flasks," "Specifications and Tolerances for Graduated Neck Type Volumetric Field Standards," "Specifications and Tolerances for Liquid Petroleum Gas and Anhydrous Ammonia Liquid Measuring Provers."

l) API Manual of Petroleum Measurement Standards, Chapter 4, *Proving Systems,* Section 3, *Small Volume Provers*, 1988; Section 4, *Tank Provers*, 1988; Section 7, *Field Standard Test Measures*, 1998, American Petroleum Institute.

2.9.4 Assuring the quality of test and calibration results

2.9.4.1 Appropriate measurement control programs should be in place and available for review for each measurement type (based upon procedures) and nominal volume range for which calibration data is provided. Note Table 4 for appropriate measurement control programs for each measurement type. Appropriate data include standard deviations and range values that represent process variation and well-characterized check standard values.

2.9.4.2 Measurement control techniques should exhibit results consistent with the procedures used to perform calibrations and should be integrated into the measurement to accurately reflect the measurement process. For those situations where statistical information is not inherent to the process, i.e., simple measurements without built-in redundancy checks, additional measurements should be made to provide experimental characterization of the measurement, sufficient for an adequate estimation of the measurement uncertainty. Those data should be available for review.

2.9.5 Accommodation and environmental conditions

2.9.5.1 To be deemed capable of making adequate measurements, calibration laboratories should provide a facility with adequate environmental controls appropriate for the level of measurements to be made, according to echelons shown in Table 2. Lower relative humidity may increase measurement error due to evaporation.

Table 2. Facility environmental control

Procedure	Temperature	Relative Humidity
I Gravimetric	20 to 23 °C, a set point ± 1 °C, maximum rate of change 1.0 °C/h	40 % to 60 % ± 10 %
II Volume Transfer	18 °C to 27 °C maximum rate of change 0.5 °C/h	40 % to 60 % ± 20 %

NOTE The environmental conditions should also be within the specifications of applicable equipment.

2.9.5.2 The environment in which testing activities are undertaken should not invalidate the results or adversely affect the required uncertainty. Particular care should be taken when such calibration practices are undertaken at sites other than the permanent laboratory facility to minimize the effects of uncontrolled environments.

2.9.5.3 Vibration, air currents, rapid temperature fluctuations, and other environmental variations should be kept to levels such that they do not diminish the measurement uncertainty of volume transfer methods or the performance of precision balances or scales when gravimetric methods are used.

2.9.5.4 The quality of water used as a calibration medium should be of adequate purity (potable) and cleanliness, and should be free from excess air entrapment. For gravimetric procedures the density should be calculated/measured to 0.00001 g/cm^3.

2.9.6 Equipment

2.9.6.1 Gravimetric

a) Mass standards used as reference standards should be traceable to a national laboratory (such as NIST) and be available at each class and range for which the laboratory is accredited, as recommended in Table 4. Sufficient historical data and uncertainty analysis should be available to support the quoted measurement uncertainties for the standards used.

b) Gravimetric methods, which generally use water as the calibration medium, require the verification of density of an adequate supply of deionized or distilled water.

c) Gravimetric methods require the use of weighing equipment with adequate accuracy and precision for the uncertainty of the measurement procedure. Appropriate control charts or range charts should be maintained to verify the measurement process.

d) Gravimetric methods require the means to adequately measure barometric air pressure, air temperature, water temperature, and relative humidity of the laboratory environment. Environmental measuring equipment should be available with the uncertainty indicated in Table 3. Relative humidity may need to be monitored more closely if evaporation is a concern.

2.9.6.2 Volumetric

a) Volume standards used as reference standards in the laboratory should be traceable to a national laboratory (such as NIST) and the laboratory should have appropriate procedures in place for verification and recalibration. The uncertainty of the national measurement institute's measurement of the primary volume standards of the laboratory should be appropriate for the uncertainty of measurement results provided.

b) Volumetric methods require appropriate temperature measurements. Environmental measuring equipment should be available with the accuracy indicated in Table 3 below. Relative humidity may need to be monitored more closely if evaporation is a concern.

Table 3. Environment measuring equipment accuracy

Procedure Type	Barometric Pressure	Temperature water/air	Relative Humidity
Gravimetric	± 135 Pa (± 1.0 mm hg)	± 0.1 °C / ± 0.5 °C	± 10% per 4 h
Volume Transfer	Not essential	± 0.5 °C	Not essential

2.9.7 Test and calibration methods and method validation

2.9.7.1 The algorithm chosen for the measurement, the reference standard to be used, and the equipment to be used for a particular calibration should be correct for that calibration. A documented procedure should be available in the laboratory to determine the correct algorithm. (Examples are provided in NBS Handbook 145, Standard Operating Procedures.)

2.9.7.2 Computer programs should have passed software quality analysis. Computer programs should be documented in detail. The documentation should include technical references that provided the basis for the algorithm, any weighing equations, and any data sets used to test the program for errors.

2.9.8 Handling of test and calibration items

2.9.8.1 The laboratory should have documented procedures to ensure adequate chain-of-custody of calibration items if required by law.

2.9.8.2 Appropriate procedures should be documented to ensure adequate tracking of calibration items that are appropriate for glass or metal volumetric standards.

2.9.9 Reporting the results

2.9.9.1 As required by ISO/IEC 17025, calibration reports should describe the volume standards with sufficient detail to avoid any ambiguity. Additional items to be included on a test report are:

a) volume,

b) uncertainty,

c) reference temperature,

d) material,

e) thermal coefficient of expansion (assumed or measured),

f) construction,

g) any identifying markings, and

h) any tolerances if appropriate.

2.9.9.2 Environmental parameters measured during the test should be provided on the test report as appropriate. These include laboratory temperature, volume standard temperature, barometric pressure and relative humidity.

2.9.9.3 Volume standards being tested should meet the appropriate classifications such as NIST, ASTM, API, or OIML, if required by laboratory customers. It is the responsibility of laboratory customers to specify the acceptable level of measurement uncertainty for their needs.

2.9.9.4 The calibration item (volume standard) should be free of any sign of abuse or damage. Signs of abuse or misuse include dents, chips, improper draining due to lack of cleanliness, and dirty sight gages. Out-of-tolerance conditions should be reported.

Table 4. Summary of technical criteria for volume calibration

Procedure	Minimum Measurement Control	Minimum Reference Standard	Minimum Traceability	Minimum Calibration Methods
I Gravimetric	Process control charts Check standards Surveillance of all standards used to provide measurement services Proficiency testing -On site assessment -Participation in interlaboratory comparisons	Appropriate mass standards ASTM Class 2 or 3; or OIML Class F_1 or F_2; or Calibrated Balance	NIST, or national level calibration periodically, based on independent historical data verification or Test of weighing equipment using correct methods of calibration and adjustment with traceable mass standards	Documented comparison calibration procedure or NIST Handbook 145, double substitution (for example) or Use of calibrated balance
II Volume Transfer	Process control charts Check standards Surveillance of selected standards Proficiency testing -On site assessment -Participation in interlaboratory comparisons	Primary volume standards with accuracy and repeatability characteristics acceptable for the type of service provided	Original NIST calibration and periodic independent verification or Calibration by accredited Echelon I laboratory, if uncertainty requirements can be met	Documented volume transfer (or water draw) procedure recognized by NIST, OIML, ASTM, or API

2.10 Hydrometer calibrations

2.10.1 Scope

This section outlines the specific technical requirements for a laboratory to be recognized as competent to carry out calibrations of hydrometers.

2.10.2 References

a) ASTM E100-95 (2001): *Standard Specification for ASTM Hydrometers*.

b) ASTM E126-92 (1998): *Standard Test Method for Inspection and Verification of Hydrometers*.

c) Hughes, J. C., "Testing of Hydrometers," *NBS Circular 555* (Washington D. C.: NBS 1954).

d) Burgess, G. K., "Standard Density and Volumetric Tables," *NBS Circular 19, 6th ed.* (Washington DC: NBS 1924).

2.10.3 Assuring the quality of test and calibration results

2.10.3.1 All sources of variability for the hydrometer calibration should be appropriately measured and monitored. Check standards should be used to ensure that the calibrations are carried out under controlled conditions. The laboratory should maintain statistical process control (SPC) commensurate with the level of uncertainty needed for the calibration. The SPC control parameters should be based on measurements of check standards (or closure parameters) and the repeatability of multiple measurements. The frequency and number of process control checks should be appropriate for the level of uncertainty claimed for the calibration.

2.10.3.2 The laboratory should have control hydrometers that adequately span the range of hydrometer materials and sizes normally calibrated by the laboratory. Every measured value of each control should be recorded and compared to its historic value to determine whether or not the process is in control. These values should be plotted on a control chart (may be done on a computer and stored electronically) that has upper and lower control limits.

2.10.4 Accommodation and environmental conditions

2.10.4.1 The environmental conditions (i.e., temperature, atmospheric pressure and relative humidity) in the hydrometer calibration area should have no more than the maximum variations permitted, depending on the materials and the level of uncertainty needed for the calibration. The reference temperature for a particular hydrometer scale may vary from 15.56 °C (15.56 °C is approximately 60 °F, which is the reference temperature for petroleum products in the United States) to 20 °C. The laboratory should have the appropriate instrumentation required to measure the environmental conditions.

2.10.4.2 The density of the water used in hydrometer calibrations should be known to within 0.000005 g/cm^3. Specific gravity is expressed as the ratio of the density of a liquid to the density of water at a specified temperature.

2.10.4.3 Vibration of equipment used in the hydrometer calibrations should be reduced to non-influential levels. If an obvious source of vibration exists, it should not adversely affect the laboratory's claimed uncertainty level.

2.10.4.4 Any laboratory that makes hydrometer comparisons should have an appropriate supply of calibration fluids with suitable surface tensions. Hydrometers should be calibrated in the liquids in which they are to be used.

2.10.4.5 Calibration liquids should be stored in an approved safety cabinet. Laboratories that make hydrometer comparisons should abide by all safety requirements set forth by a regulatory counsel, (e.g., chemical labeling, EPA and OSHA guidelines, etc.).

2.10.5 Equipment

2.10.5.1 The laboratory should have the appropriate equipment required to perform hydrometer calibrations at the uncertainty level for which it is accredited. All equipment should be properly maintained.

2.10.5.2 The laboratory that performs hydrometer comparisons should have master hydrometers for which the calibrations are directly traceable to the appropriate national standards laboratory. The appropriate calibration corrections to these master hydrometers should be applied.

2.10.5.3 The laboratory should have the equipment needed to make auxiliary measurements of hydrometers, (e.g., balances, mass standards, knowledge of water density, etc.).

2.10.5.4 Any laboratory that makes hydrometer comparisons should abide by all safety requirements set forth by a regulatory counsel, (e.g., chemical labelling, EPA and OSHA guidelines, etc.).

2.10.5.5 The laboratory should have temperature measuring capabilities suitable for the calibration procedure. In the case of measuring the specific gravity of a liquid with a master hydrometer, temperature measurement of the liquid accurate to ±0.01 °C is required.

2.10.5.6 A laboratory that makes hydrometer comparisons should have a ventilated chemical hood to exhaust any harmful fumes from the working area.

2.10.6 Test and calibration methods and method validation

2.10.6.1 The wide use of hydrometers for many different purposes has led to various stem scales for unique applications (e.g., specific gravity, percentage alcohol, degrees API, degrees Baume and Brix). The appropriate stem scale should be evaluated so that the appropriate calibration procedure can be selected and performed for the expected use of hydrometer.

2.10.6.2 Ideally, hydrometers under test are compared directly to master hydrometers in the kinds of liquids in which they are to be used. This comparison is performed in a clear, smooth glass cylinder of suitable size. The calibration liquid should be well stirred before each comparison to minimize temperature gradients in the liquid.

2.10.6.3 The laboratory should have a manual detailing the procedures to be followed for each type of hydrometers being calibrated. This manual should contain all pertinent information needed for calibration to the level of uncertainty for which it is accredited.

2.10.7 Handling of test and calibration items

2.10.7.1 Hydrometers should be cleaned and stored in a manner that prevents accidental contact with materials which could damage its surfaces. Since hydrometers are made of glass and can be easily broken, they should be handled only by an experienced operator.

2.10.7.2 Inspection should be made of all hydrometers to be calibrated for bent stems, twisted scales and loose material inside the body of the hydrometer.

2.10.7.3 The hydrometer should be wiped with alcohol and dried to assure a clean surface before it is immersed in the calibration liquid.

2.10.8 Reporting the results

2.10.8.1 As required by ISO/IEC 17025, calibration reports should describe the hydrometer with sufficient detail to avoid any ambiguity.

2.10.8.2 The uncertainty for the hydrometer should be derived from a model of the measurement system that includes, as applicable, the uncertainties due to:

a) master hydrometer,

b) long term reproducibility of the measurement system,

c) thermal expansion, and,

d) other appropriate factors.

2.10.8.3 A historical registry should be kept for all control hydrometers (see 2.10.3.2).

2.11 Rockwell hardness

2.11.1 Scope

This section of the handbook provides technical criteria needed to assess the competence of a calibration agency that performs Rockwell hardness calibrations. The section is organized beginning with General Technical Criteria that applies to all categories of Rockwell hardness calibration services, followed by Specific Technical Criteria for the specific types of Rockwell hardness calibration services.

2.11.2 General technical criteria for Rockwell hardness calibration agencies

2.11.2.1 Introduction

a) **Requirements and recommendations:** Hardness standards are derived standards based on the method of measurement. The standards used for most other parameters covered by NIST Handbook 150-2, hereafter referred to as the Guide, differ in that they may be traced to measurement artifacts, scientific principles, or defined by physical phenomena. Because Rockwell hardness is more of a procedural standard, the technical guide must be more prescriptive and therefore the requirements that may be only recommendations or indications of good practice in other sections become rigid requirements when

considering hardness calibrations. This section therefore uses the terms "shall" and "must" to indicate an absolute requirement in the same way that they are used in NIST Handbook 150. Less stringent requirements are indicated by the use of terms like "should" or "may."

b) Under this accreditation program, laboratories are assessed for competency in performing procedures defined as ***calibrations***. In the case of Rockwell hardness, U.S. national and international test standards use both ***calibration*** and ***verification*** terminology to describe the certification procedures for Rockwell hardness machines, test blocks, and indenters. In the case of test blocks and indenters, these procedures also are referred to as ***standardizations***. The requirements outlined in this section of the Guide include all of these procedures as part of the calibration process, and thus are subject to accreditation. The term ***calibration*** will be used as a general term that will include ***calibration, verification*** and ***standardization*** as they pertain to Rockwell hardness.

c) **Calibration laboratory and calibration agency:** In most cases, the calibrations discussed in the other sections of this Guide are conducted at laboratory facilities that are maintained at permanent sites. This Guide uses ***calibration laboratory*** as the designation for these laboratories. Calibrations of Rockwell hardness machines are often conducted "in the field" at the location where the hardness machine is used. In this case, the term ***calibration laboratory*** is inappropriate as a description of the calibration activity. Consequently, in this section of the Guide, the term ***calibration agency*** is used as the designation for the entity conducting Rockwell hardness calibrations.

d) For purposes of accreditation, the calibration agencies that perform Rockwell hardness calibrations have been divided into four categories which provide services as follows:

1) **Direct verification of Rockwell hardness machines**: A process for verifying that critical components of the hardness machine are within allowable tolerances by directly verifying specified parameters. These parameters may include test force application, the depth measuring system, and machine hysteresis.

2) **Indirect verification of Rockwell hardness machines**: A process for verifying that the measurement performance of the hardness machine is within allowable tolerances by measuring the Rockwell hardness of standardized test blocks.

3) **Standardization of Rockwell hardness test blocks**: A process for verifying the geometrical and physical parameters of the test block, and calibrating the test block hardness with respect to a stated reference standard.

4) **Standardization of Rockwell hardness indenters**: A process for verifying the geometrical and physical parameters of the indenter, and calibrating the indenter performance by comparison measurements with a standardizing indenter.

e) **Rockwell and Rockwell Superficial hardness tests:** The test principles, testing procedures and verification procedures are essentially identical for both the Rockwell and Rockwell Superficial hardness tests. The significant differences between the two tests are that both the preliminary and total applied test forces are smaller for the Rockwell Superficial test than for the Rockwell test. The same type and size indenters may be used for either test, depending on the scale being employed. Although some tolerance values may be different, the basic criteria for assessing calibration agencies are, for the most part, the same for both Rockwell and Rockwell Superficial hardness testing. Therefore, henceforth in this section, the term Rockwell will imply both Rockwell and Superficial Rockwell unless otherwise stated.

f) The technical criteria are based on requirements as stated in the U.S. national standards published by ASTM, international standards published by ISO, and good laboratory practices recognized by NVLAP and the U.S. hardness community. The Rockwell hardness calibration agency is required to meet each of the General Technical Criteria requirements and the Specific Technical Criteria requirements.

g) Traceability of Rockwell hardness calibration measurements require a linkage to nationally recognized reference standards having documented uncertainties. Therefore, when hardness measurement is a part of the calibration process, a calibration agency must state to which Rockwell reference standard or standards the calibration is traceable. For example, the calibration values may be traceable to U.S. national Rockwell standards, the national Rockwell standards of another country, or commercial reference standards. The calibration agency may perform separate calibrations referencing more than one reference standard; however, the specific standards that each calibration is traceable to must be stated. In cases where U.S. national Rockwell standards exist, and there is a mechanism available to transfer the U.S. national reference standard values, the calibration agency must be capable of referencing its calibrations to the U.S. national scales.

h) **ASTM or ISO test standard:** At the time of this publication, the U.S. national Rockwell hardness test standard published by ASTM and the international Rockwell hardness test standards published by ISO are not in complete agreement on the requirements for Rockwell hardness calibrations. Consequently, the calibration agency must specify the Rockwell hardness test method standards to which accreditation is desired. The calibration agency may obtain accreditation to both ASTM and ISO test standards. The most recently published book version of the ASTM test methods or the most recently published ISO test methods shall always be used for a laboratory evaluation, depending on the test method or methods for which the calibration agency has requested accreditation.

i) The requirements of the applicable test methods of ASTM and/or ISO provide the basis for the criteria given here, but additional criteria not contained in either the ASTM or ISO test methods, but considered essential for good laboratory practice for calibration agencies, may also be given.

2.11.2.2 References

a) **ASTM Standards**

ASTM E 18 (current version), *Standard Test Methods for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials.*

b) **ISO Standards**

1) ISO 6508-1 (current version), *Metallic materials - Rockwell hardness test - Part 1: Test method (scales A, B, C, D, E, F, G, H, K, N, T).*
(Replaces ISO 6508:1986 and ISO 1024:1989)

2) ISO 6508-2 (current version), *Metallic materials - Rockwell hardness test - Part 2: Verification and calibration of testing machines (scales A, B, C, D, E, F, G, H, K, N, T).*
(Replaces ISO 716:1986 and ISO 1079:1989)

3) ISO 6508-3 (current version), *Metallic materials - Rockwell hardness test - Part 3: Calibration of reference blocks (scales A, B, C, D, E, F, G, H, K, N, T).*
(Replaces ISO 674:1988 and ISO 1355:1989)

c) **General**

Low, Samuel R., NIST Recommended Practice Guide "Rockwell Hardness Measurement of Metallic Materials," *NIST Special Publication 960-5*, 2001.

2.11.2.3 Definitions

a) **Rockwell hardness machines**

1) **Repeatability:** How well a Rockwell hardness machine can repeat the same measurement value on a perfectly uniform test sample when there is a relatively short time interval between measurements.

2) **Reproducibility:** How much a Rockwell hardness machine varies in its measurement results over an extended time interval.

3) **Standardizing machine:** A Rockwell hardness machine used for the standardization of Rockwell hardness indenters, and for the standardization of Rockwell hardness test blocks. The standardizing machine differs from a regular Rockwell hardness testing machine by having tighter tolerances on certain parameters.

4) **Testing machine:** A Rockwell hardness machine used for general testing purposes.

b) **Rockwell indenters**

1) **Primary standardizing indenter:** The primary national indenter used for the standardization of primary standardized test blocks by the national Rockwell hardness standardization laboratory (NIST in the U.S.).

2) **Standardizing indenter:** Intended to be used for the indirect verification of Rockwell standardizing machines, for the standardization of testing indenters, and for the standardization of test blocks. Standardizing indenters have tighter tolerances than testing indenters for geometrical features and/or performance.

3) **Testing indenter:** Intended for every day use with Rockwell testing machines, and for the indirect verification of Rockwell testing machines.

4) **Rockwell regular scale diamond indenter:** A Rockwell spheroconical diamond indenter certified for use only on the regular Rockwell scales that use a diamond indenter (i.e., HRA, HRC, and HRD).

5) **Rockwell superficial scale diamond indenter:** A Rockwell spheroconical diamond indenter certified for use only on the Rockwell superficial scales that use a diamond indenter (i.e., HR15N, HR30N, and HR45N).

6) **Rockwell combination diamond indenter:** A Rockwell spheroconical diamond indenter certified for use only on all Rockwell scales that use a diamond indenter (i.e., HRA, HRC, HRD, HR15N, HR30N, and HR45N).

c) **Rockwell test blocks**

1) **Monitoring test blocks:** Test blocks used to monitor the daily performance of a Rockwell standardizing machine.

2) **Primary standardized test blocks:** Standardized test blocks that have been certified by the primary national hardness standardizing laboratory (NIST in the U.S.) traceable to the national hardness standards.

3) **Standardized test blocks:** Test blocks that have been calibrated traceable to specific Rockwell hardness standards.

d) **Testing cycle**: The sequence of applying the test forces during the Rockwell test. The cycle of applying the forces may be described by force variations with time.

2.11.2.4 Scope of accreditation

a) The calibration agency may seek a separate accreditation for one or more of these categories of calibration services:

1) direct verification of Rockwell hardness machines,

2) indirect verification of Rockwell hardness machines,

3) standardization of Rockwell hardness test blocks, and/or

4) standardization of Rockwell hardness indenters.

b) The Rockwell scales for which NVLAP accreditation is provided are given in Table 1. A calibration agency may request accreditation for any or all of these scales.

Table 1. Specific Rockwell scales and indenters used

REGULAR ROCKWELL SCALES [Preliminary Test Force of 10 kgf (98.1 N)]			ROCKWELL SUPERFICIAL SCALES [Preliminary Test Force of 3 kgf (29.4 N)]		
Scale	**Total Test Force**	**Indenter[4]**	**Scale**	**Total Test Force**	**Indenter[4]**
HRA	60 kgf (588.4 N)	Spheroconical Diamond	HR15N	15 kgf(147 N)	Spheroconical Diamond
HRB	100 kgf (980.7 N)	Ball - 1/16 in. (1.588 mm)	HR30N	30 kgf (294 N)	Spheroconical Diamond
HRC	150 kgf (1471 N)	Spheroconical Diamond	HR45N	45 kgf (441 N)	Spheroconical Diamond
HRD	100 kgf (980.7 N)	Spheroconical Diamond	HR15T	15 kgf (147 N)	Ball - 1/16 in. (1.588 mm)
HRE	100 kgf (980.7 N)	Ball - 1/8 in. (1.175 mm)	HR30T	30 kgf (294 N)	Ball - 1/16 in. (1.588 mm)
HRF	60 kgf (588.4 N)	Ball - 1/16 in. (1.588 mm)	HR45T	45 kgf (441 N)	Ball - 1/16 in. (1.588 mm)
HRG	150 kgf (1471 N)	Ball - 1/16 in. (1.588 mm)	HR15W	15 kgf (147N)	Ball - 1/8 in. (3.175 mm)
HRH	60 kgf (588.4 N)	Ball - 1/8 in. (3.175 mm)	HR30W	30 kgf (294 N)	Ball - 1/8 in. (3.175 mm)

REGULAR ROCKWELL SCALES [Preliminary Test Force of 10 kgf (98.1 N)]			ROCKWELL SUPERFICIAL SCALES [Preliminary Test Force of 3 kgf (29.4 N)]		
HRK	150 kgf (1471 N)	Ball - 1/8 in. (3.175 mm)	HR45W	45 kgf (441 N)	Ball - 1/8 in. (3.175 mm)
HRL	60 kgf (588.4 N)	Ball - 1/4 in. (6.350 mm)	HR15X	15 kgf (147 N)	Ball - 1/4 in. (6.350 mm)
HRM	100 kgf (980.7 N)	Ball - 1/4 in. (6.350 mm)	HR30X	30 kgf (294 N)	Ball - 1/4 in. (6.350 mm)
HRP	150 kgf (1471 N)	Ball - 1/4 in. (6.350 mm)	HR45X	45 kgf (441 N)	Ball - 1/4 in. (6.350 mm)
HRR	60 kgf (588.4 N)	Ball - 1/2 in. (12.70 mm)	HR15Y	15 kgf (147 N)	Ball - 1/2 in. (12.70 mm)
HRS	100 kgf (980.7 N)	Ball - 1/2 in. (12.70 mm)	HR30Y	30 kgf (294 N)	Ball - 1/2 in. (12.70 mm)
HRV	150 kgf (1471 N)	Ball - 1/2 in. (12.70 mm)	HR45Y	45 kgf (441 N)	Ball - 1/2 in. (12.70 mm)

[4] The measurement given is the ball diameter.

2.11.2.5 Test and calibration methods and method validation

a) Both the principle of the test and the procedures to be followed when performing Rockwell hardness tests as part of the calibration process are described in the appropriate ASTM or ISO Rockwell test method standards (see References section). These procedures shall be followed, unless otherwise noted, for all Rockwell testing.

b) The procedures to be followed when performing verifications, standardizations, and calibrations are described in the appropriate ASTM or ISO Rockwell test method standards (see References section). These procedures shall be followed, unless otherwise noted.

2.11.2.6 Personnel

Personnel operating or verifying the testing equipment or analyzing verification or test results shall be able to demonstrate detailed knowledge of the appropriate test method(s) as well as the operation of the testing equipment.

2.11.2.7 Accommodation and environmental conditions

a) **Temperature and Humidity:** When specified by the relevant test standard, the temperature and relative humidity of the site where Rockwell hardness calibrations are conducted shall be within the ranges specified for the type of hardness machine (standardizing or testing). The temperature during the verifications shall be recorded.

b) The calibration area shall have adequate lighting and clearance for efficient testing and verifications.

c) The calibration area shall be clean and free of dust and corrosive vapors.

d) All Rockwell standardizing machines shall be in isolated locations that are free from shock or vibration that could alter the calibration of the hardness machines or affect hardness measurements during calibration of test blocks or indenters.

e) Rockwell hardness machines and verification devices shall be protected from electrical interference that may affect results.

2.11.2.8 Document control

The most recently published book version of the ASTM Rockwell test method standard and/or the most recently published applicable ISO test methods shall be immediately available to all applicable personnel of the calibration agency.

2.11.2.9 Equipment

a) All instruments and devices used to make measurements as part of a calibration shall have been calibrated traceable to United States national standards, where a system of traceability exists. Current certificates indicating calibration traceability shall be available.

b) All instruments and devices used to make measurements as part of a calibration shall have uncertainties as required by the relevant test standard.

c) In cases where temperature and/or humidity measurements are required by the test standard, but accuracy requirements for the measuring instruments are not specified, the accuracy of the instruments shall be at least:

   1) **Temperature**
   ±0.5 °C for a standardizing machine
   ±1 °C for a testing machine

   2) **Humidity**
   ±10 % relative humidity.

2.11.3 Direct verification of Rockwell hardness machines: specific technical criteria for Rockwell hardness calibration agencies

2.11.3.1 Scope

This section contains specific technical criteria that a calibration agency shall meet in order to be accredited to carry out **direct verifications** of Rockwell hardness machines as specified in the relevant test standards. These criteria are in addition to the general criteria listed above in the General Technical Criteria for Rockwell Hardness Calibration Agencies section.

2.11.3.2 Definition

Direct verification of a Rockwell hardness machine is a process for verifying that critical components of the hardness machine are within allowable tolerances by directly verifying specified parameters. These parameters include test force application, the depth measuring system, and machine hysteresis.

NOTE Current versions of ASTM and ISO test method standards may not include requirements for measuring machine hysteresis, but it is likely that future revisions of the ASTM standard will include this requirement.

2.11.3.3 Categories of direct verifications

a) This section covers calibration agencies conducting direct verifications within their own on-site calibration facility, and calibration agencies conducting direct verifications at off-site facilities outside the control of the calibration agency.

1) **On-site direct verifications**: Direct verifications conducted within a calibration agency's own calibration laboratory will be termed "on-site direct verifications." An on-site direct verification includes both the verification of hardness machines to be used within the calibration laboratory and the verification of newly manufactured or repaired hardness machines that will be directly verified at the calibration laboratory then transported to the customer.

2) **Off-site direct verifications**: Direct verifications conducted at facilities outside the control of the calibration agency will be termed "off-site direct verifications," even in cases where the verifications are conducted in a controlled laboratory environment. An off-site direct verification usually involves transporting the verification instruments and reference materials to the location where the hardness machine is used.

b) **Standardizing and testing machines**: This section covers two categories of hardness machines:

1) standardizing machines used for the standardization of test blocks and indenters, and

2) testing machines for general-use purposes.

The criteria are similar for both categories, but in many cases, tolerances are tighter for standardizing machines than for testing machines.

c) **Rockwell and Rockwell Superficial**: This section covers both Rockwell and Rockwell Superficial hardness machines. It is necessary to differentiate between the direct verifications of these types of Rockwell machines because of the differing requirements of verification equipment.

2.11.3.4 Scope of accreditation: direct verification

a) The scope of accreditation for direct verification is independent of individual Rockwell hardness scales, but rather is based on the categories of hardness machines and categories of direct verifications. The calibration agency's scope of accreditation will state the categories of hardness machines and the categories of direct verification for which the calibration agency is accredited as indicated in Table 2.

b) To obtain accreditation for conducting a direct verification of Rockwell hardness machines, the calibration agency shall be capable of verifying or calibrating each parameter as specified by the relevant test standard.

Table 2. The scope of accreditation for calibration agencies accredited to conduct direct verifications

CATEGORIES OF HARDNESS MACHINES
Rockwell Standardizing Machine
Rockwell Superficial Standardizing Machine
Rockwell Testing Machine
Rockwell Superficial Testing Machine
CATEGORIES OF DIRECT VERIFICATIONS
On-site Direct Verifications
Off-site Direct Verifications
INDIVIDUAL VERIFICATIONS
Verification of the test forces
Verification of the depth measuring system
Verification of machine hysteresis

c) Current versions of ASTM and ISO test method standards may not require periodic direct verification of Rockwell hardness machines following the initial direct verification. It is recognized that a calibration agency may desire to be accredited to conduct one or more of the verifications required by a direct verification without being accredited for a complete direct verification. Under this program, a calibration agency may obtain separate accreditations for verifying single parameters of Rockwell hardness machines. In this case, the calibration agency will be accredited for conducting the individual verifications only, and will not be accredited for conducting a complete direct verification under the scope of accreditation. Accreditation for conducting individual verifications of Rockwell hardness machines is offered for the following parameters:

1) verification of the test forces,

2) verification of the depth measuring system, and

3) verification of machine hysteresis as given in Table 2.

d) The scope of accreditation must reflect the categories of hardness machines to which each of these individual verifications apply.

2.11.3.5 Accommodation and environmental conditions

a) **On-site**: A calibration agency conducting on-site direct verifications shall control the environment where the hardness machine is located as specified in the General Technical Criteria for Rockwell Hardness Calibration Agencies section.

b) **Off-site**: A calibration agency conducting off-site direct verifications usually does not have control over the environment where the hardness machine is located. It is the responsibility of the calibration agency to determine whether the environment is suitable for conducting the direct verifications.

2.11.3.6 Equipment

a) A calibration agency conducting off-site direct verifications shall have appropriate instrumentation and reference materials that are transportable.

b) **Temperature and humidity**: The calibration agency shall have appropriate instruments, as required, for the measurement and recording of the temperature and humidity.

c) **Verification of the test forces**: The calibration agency shall have suitable instruments for the measurement of the test forces. The force measuring instruments shall be capable of measuring the test forces as the forces are applied during a Rockwell hardness test (extended dwell times are allowed). Measurements from the force measuring instruments shall have the appropriate uncertainty, as specified in the relevant test method standards, for each test force to be measured.

d) **Verification of the depth measuring system**: The calibration agency shall have suitable instruments or reference materials for the verification of the depth measuring system. The verification method shall have the appropriate measurement uncertainty, as specified in the relevant test method standards.

e) **Verification of machine hysteresis**: The hysteresis of the hardness machine is verified by means of a device or system that will allow the hardness machine to perform a Rockwell test without causing any measurable permanent deformation in a test piece. Such a device can be a blunt indenter or the indenter holder acting directly against the anvil or a very hard test piece. The calibration agency shall have an appropriate system and procedures to conduct this measurement.

2.11.3.7 Test and calibration methods and method validation

a) In general, the calibration agency shall follow the calibration and verification requirements and procedures specified in the relevant ASTM and ISO test method standards. The calibration agency shall have documented procedures for conducting each of the direct verification procedures.

b) In the absence of requirements for the verification of machine hysteresis, the following procedures are recommended. At least five Rockwell hardness tests shall be performed using a blunt indenter or the indenter holder acting directly against the anvil or a very hard test piece using the highest test force to be used on this machine. The goal is to perform a purely elastic test that results in no permanent indentation. This permits the determination of the hysteresis in the flexure of the machine.

2.11.3.8 Assuring the quality of test and calibration results

Each direct verification measurement must have an uncertainty small enough for the purpose. The uncertainty is to be calculated in a manner consistent with NIST Technical Note 1297.

2.11.3.9 Reporting the results

The certificate and standardization report shall be prepared by the person conducting the verification and shall include all information as required by NIST Handbook 150 and the relevant test standard.

2.11.4 Indirect verification of Rockwell hardness machines: specific technical criteria for Rockwell hardness calibration agencies

2.11.4.1 Scope

a) This section contains specific technical criteria that a calibration agency shall meet in order to be accredited to carry out indirect verifications of Rockwell hardness machines as specified in the relevant test standards. These criteria are in addition to the general criteria listed above in the General Technical Criteria for Rockwell Hardness Calibration Agencies section.

b) **Indirect verification** of a Rockwell hardness machine is a process for verifying that the measurement performance of the hardness machine is within allowable tolerances by measuring standardized test blocks. The indirect verification requirements stated in Rockwell test standards may require that the verifications be made using both the calibration agency's indenter and the customer's indenter(s). The calibration agency must state to what Rockwell reference standard or standards the calibration measurements are traceable.

c) **Categories of indirect verifications**: This section covers calibration agencies conducting indirect verifications within their own calibration facility, and calibration agencies conducting indirect verifications at off-site facilities outside the control of the calibration agency.

   1) **On-site indirect verifications**: Indirect verifications conducted within a calibration agency's own calibration facility will be termed "on-site indirect verifications."

   2) **Off-site indirect verifications**: Indirect verifications conducted at facilities outside the control of the calibration agency will be termed "off-site indirect verifications," even in cases where the verifications are conducted in a controlled laboratory environment. An off-site indirect verification usually involves transporting the verification reference materials to the location where the hardness machine is used.

d) **Standardizing and testing machines**: This section covers two categories of hardness machines:

   1) standardizing machines used for the standardization of test blocks and indenters, and

   2) testing machines for non-standardizing or general-use purposes.

   The criteria are similar for both categories, but in some cases, tolerances are tighter for standardizing machines than for non-standardizing machines.

2.11.4.2 Scope of accreditation: indirect verification

The scope of accreditation for indirect verification is based on the categories of hardness machines and categories of indirect verification. The scope of accreditation is valid for all Rockwell scales unless the calibration agency does not possess or cannot demonstrate the availability of reference test blocks for all Rockwell scales, or the calibration agency does not possess appropriate indenters for all Rockwell scales. The calibration agency's scope of accreditation will state the categories of hardness machines and the categories of indirect verification that the calibration agency is accredited to conduct as indicated in Table 3. The scope of accreditation will state the Rockwell scales that are covered.

Table 3. The scope of accreditation for calibration agencies accredited to conduct indirect verifications

CATEGORIES OF HARDNESS MACHINES
Rockwell Standardizing Machine
Rockwell Testing Machine
CATEGORIES OF INDIRECT VERIFICATIONS
On-site Indirect Verifications
Off-site Indirect Verifications

2.11.4.3 Accommodation and environmental conditions

a) **On-site**: A calibration agency conducting on-site indirect verifications shall control the environment where the hardness machine is located as specified in the General Technical Criteria for Rockwell Hardness Calibration Agencies section.

b) **Off-site**: A calibration agency conducting off-site indirect verifications usually does not have control over the environment where the hardness machine is located. It is the responsibility of the calibration agency to determine whether the environment is suitable for conducting the indirect verifications.

2.11.4.4 Equipment

a) A calibration agency conducting off-site indirect verifications shall have appropriate instrumentation and reference materials that are transportable.

b) **Temperature and humidity:** The calibration agency shall have appropriate instruments, as required by the relevant test standard, for the measurement and recording of the temperature and humidity.

c) The calibration agency shall maintain an inventory of appropriate standardized test blocks and indenters needed to conduct the indirect verification in accordance with the relevant ASTM and ISO test method standards. Each test block and indenter shall have been calibrated by a calibration agency accredited under the requirements of this Guide.

   1) **Standardized test blocks:** The calibration agency shall maintain an inventory of appropriate standardized test blocks in the required ranges of each Rockwell scale that the calibration agency ordinarily conducts indirect verifications. The standardized test blocks shall state traceability to specific Rockwell reference standards. If the calibration agency's current inventory of standardized test blocks does not include some of the less commonly used Rockwell scales, this will not prevent accreditation for standardizing all Rockwell scales. In this case, the calibration agency shall provide evidence that it has access to a supplier that can supply appropriate standardized blocks for each Rockwell scale.

   2) **Indenters:** The calibration agency shall maintain an inventory of appropriate Rockwell indenters as specified by the relevant test standard. Indenters shall be available for each Rockwell scale covered by the scope of accreditation.

2.11.4.5 Test and calibration methods and method validation

In general, the calibration agency shall follow the indirect verification requirements and procedures specified in the relevant test method standards. Indirect verification involves verifying the performance of the test machine by means of standardized test blocks and indenters. The calibration agency shall have documented procedures for conducting indirect verifications.

2.11.4.6 Assuring the quality of test and calibration results

a) Indirect verification is an investigation of machine and indenter bias. This bias measurement may be masked by:

   1) standardized block uncertainty,

   2) repeatability of the hardness machine,

   3) reproducibility of the hardness machine.

b) Statements of uncertainty shall be consistent with NIST Technical Note 1297.

2.11.4.7 Reporting the results

The certificate and standardization report shall be prepared by the person conducting the verification and shall include all information as required by NIST Handbook 150 and the relevant test standard.

2.11.5 Standardization of Rockwell hardness test blocks: specific technical criteria for Rockwell hardness calibration agencies

2.11.5.1 Scope

This section contains specific criteria that a calibration agency must meet in order to be accredited to standardize Rockwell hardness test blocks as specified in the relevant test standards. These criteria are in addition to the general criteria listed above in the General Technical Criteria for Rockwell Hardness Calibration Agencies section.

2.11.5.2 Scope of accreditation

The Scope of Accreditation for agencies standardizing Rockwell hardness test blocks covers all Rockwell and Rockwell Superficial scales listed in Table 1 of the General Technical Criteria for Rockwell Hardness Calibration Agencies section. A calibration agency may request accreditation for any or all of these scales.

2.11.5.3 Accommodation and environmental conditions

The calibration agency shall control the environment where the standardizing machine is located as specified in the General Technical Criteria for Rockwell Hardness Calibration Agencies section.

2.11.5.4 Equipment

a) **Temperature and humidity:** The calibration agency shall have suitable instruments, as required, for the measurement and recording of the temperature and humidity.

b) **Standardizing test machine:** The calibration agency shall have one or more standardizing test machines which are capable of performing measurements of each type of Rockwell hardness scale listed under the calibration agency's scope of accreditation. One standardizing test machine may be used for the standardization of test blocks of multiple Rockwell hardness scales. Each standardizing machine shall have been verified by a calibration agency accredited under the requirements of this Guide. Verified standardizing machines shall have a sticker or tag attached indicating the date and the extent of the verification.

c) **Time:** In cases where the standardizing test machine does not control the timing aspects of the standardization test cycle, the calibration agency shall have an appropriate timing device having a valid certificate of calibration.

d) **Standardizing indenters:** The calibration agency shall have one or more standardizing indenters appropriate for each hardness scale listed under the calibration agency's scope of accreditation. Each standardizing indenter shall have been verified by a calibration agency accredited under the requirements of this Guide. All test block standardizations shall be made using these standardizing indenters.

e) **Monitoring test blocks:** The calibration agency shall maintain an inventory of monitoring test blocks in the required ranges for each Rockwell scale that the calibration agency conducts hardness measurements as part of the test block standardization procedures.

f) **Flatness, parallelism, and surface roughness:** The calibration agency shall have appropriate equipment needed to make measurements of the test block flatness, parallelism, and surface roughness, in accordance with applicable standards. This equipment shall have valid certificates of calibration.

2.11.5.5 Test and calibration methods and method validation

a) The calibration agency shall have documented procedures for measuring the geometrical and physical requirements (thickness, test surface area, deviation from flatness, deviation from parallelism, and surface roughness) of the test blocks. The calibration agency shall demonstrate a suitable sampling plan for ensuring the geometrical and physical requirements are met for all test blocks.

b) In general, the calibration agency shall follow the standardization requirements and procedures specified in the relevant test method standards. The calibration agency shall have documented procedures for conducting the standardizations of test blocks.

c) **Calibration corrections:** As a result of the indirect verification of a standardizing machine and indenter, a measurement bias is likely to be found with respect to the certified values of the standardized reference test blocks that were used. When allowed by the relevant test method standards, the calibration agency may desire to mathematically correct subsequent measurements for this bias,. If such corrections are made, the calibration agency shall have documented procedures for determining and making these corrections, and shall demonstrate the validity of the corrections.

2.11.5.6 Assuring the quality of test and calibration results

a) The calibration agency shall monitor the performance of the standardizing machine between indirect verifications. The monitoring procedures shall be in accordance with the appropriate test method standard when requirements are stated. The monitoring procedures should involve measuring monitoring test blocks each day that standardizations are made, and tracking the performance of the

standardizing machine using control charts. Control charts are intended to indicate a loss of measurement control in the performance of the standardizing machine.

b) Estimates of uncertainty of measurements should include all components that are evaluated by statistical methods (type A), and all components that are evaluated by other methods (type B). Sources of uncertainty in the calibration of standardized test blocks include, but may not be limited to:

   1) repeatability of standardizing machine,

   2) day-to-day reproducibility of standardizing machine,

   3) standardized block non-uniformity in hardness,

   4) uncertainty in fitting calibration curve and correction, if used,

   5) machine and indenter error if a calibration curve is not used.

c) Statements of uncertainty shall be consistent with NIST Technical Note 1297.

2.11.5.7 Handling of test and calibration items

a) Prior to standardization, the test blocks shall be placed in the test environment, and allowed sufficient time to reach thermal equilibrium.

b) Prior to and after standardization, the test blocks shall be stored in a manner that will prevent mechanical damage such as dents and scratches, or corrosive damage such as rust or oxidation.

c) To minimize thermal changes and corrosion of the test blocks, unnecessary contact with bare hands should be avoided.

d) The standardized test blocks should be packaged in a manner that adequately protects against mechanical and corrosive damage.

2.11.5.8 Records

Records must be kept for each test block that is calibrated. These records must be in accordance with applicable test method standards, but should include the following:

a) block serial number,

b) block manufacturer,

c) date of manufacture and/or date of purchase,

d) date of standardization,

e) standardizing machine serial number,

f) standardizing indenter serial number,

g) indentation measuring instrument manufacturer and serial number (for applicable hardness methods),

h) personnel making measurements (block geometry and hardness),

i) standardization test cycle (dwell times and force application times or velocities),

j) standardization temperature,

k) applicable test method standard,

l) all standardization measurement values,

m) certified hardness value,

n) uncertainty in the certified hardness value, and

o) the Rockwell reference standard to which the certified hardness value is traceable.

2.11.5.9 Reporting the results

The certificate and standardization report shall be prepared by the person conducting the standardization and shall include all information as required by NIST Handbook 150 and the relevant test standard.

2.11.6 Standardization of Rockwell hardness indenters: specific technical criteria for Rockwell hardness calibration agencies

2.11.6.1 Scope

a) This section contains specific criteria that a calibration agency must meet in order to be accredited to standardize Rockwell hardness indenters as specified in the relevant test standards. These criteria are in addition to the general criteria listed above in the General Technical Criteria for Rockwell Hardness Calibration Agencies section. Accreditation covers the procedures to standardize new Rockwell hardness indenters and the procedures for the periodic verification of the geometry and performance of Rockwell indenters. The procedures are essentially identical for both procedures.

b) **Standardizing and testing indenters:** This section covers two categories of indenters:

   1) **Standardizing indenters** - intended to be used for the indirect verification of Rockwell standardizing machines, for the standardization of testing indenters, and for the standardization of test blocks, and

   2) **Testing indenters** - intended for general-use purposes with Rockwell testing machines, and for the indirect verification of Rockwell testing machines.

   The criteria are similar for both categories, but standardizing indenters usually have tighter tolerances than testing indenters for geometrical features and performance.

2.11.6.2 Scope of accreditation

a) The scope of accreditation for the standardization of indenters is based on the categories and two classes of Rockwell and Rockwell Superficial indenters. The scope of accreditation covers all Rockwell indenters listed in Table 1 of the General Technical Criteria for Rockwell Hardness Calibration Agencies section. A calibration agency may request accreditation for any or all of these indenters. The calibration agency's scope of accreditation will state the categories and classes of indenters for which the calibration agency is accredited to conduct standardizations and periodic verifications, as indicated in Table 4.

b) **Verification of Rockwell diamond indenters:** In the case of Rockwell spheroconical diamond indenters, verification of the performance of the indenter and verification of the geometrical and physical attributes of the indenter are separate accreditations.

c) **Standardizing indenters:** Standardization of standardizing indenters may be conducted by a calibration agency only when this function is not available from the national standardizing laboratory (NIST in the U.S.). When standardizing indenters are available from the national standardizing laboratory, a calibration agency's accreditation for this function is no longer valid.

Table 4. The scope of accreditation for calibration agencies accredited to conduct standardizations of Rockwell Indenters

CATEGORIES OF ROCKWELL DIAMOND INDENTERS
Rockwell Regular Scale Diamond - Performance Verification
Rockwell Superficial Scale Diamond - Performance Verification
Rockwell Combination Diamond - Performance Verification (Rockwell and Rockwell Superficial)
Rockwell Diamond Indenter - Geometrical Verification (Rockwell and Rockwell Superficial)
CATEGORIES OF ROCKWELL BALL INDENTERS
Ball - 1/16 in. (1.588 mm) diameter
Ball - 1/8 in. (3.175 mm) diameter
Ball - 1/4 in. (6.350 mm) diameter
Ball - 1/4 in. (6.350 mm) diameter
Ball - 1/2 in. (12.70 mm) diameter
CLASSES OF ROCKWELL HARDNESS INDENTERS
Rockwell Standardizing Indenter
Rockwell Testing Indenter

2.11.6.3 Accommodation and environmental conditions

The calibration agency shall control the environment where the standardizing machine is located as specified in the General Technical Criteria for Rockwell Hardness Calibration Agencies section.

2.11.6.4 Equipment

a) **Temperature and humidity:** The calibration agency shall have appropriate instruments, as required, for the measurement and recording of the temperature and humidity.

b) **Standardizing test machine:** The calibration agency shall have one or more standardizing test machines which are capable of performing measurements of each Rockwell hardness scale required for the standardization of each type and category of indenter listed under the calibration agency's scope of accreditation. One standardizing test machine may be used for multiple Rockwell hardness scales. Each standardizing machine shall have been verified by a calibration agency accredited under the requirements of this Guide. Verified standardizing machines shall have a sticker or tag attached indicating the date and the extent of the verification.

c) **Time:** In cases where the standardizing test machine does not control the timing aspects of the calibration test cycle, the calibration agency shall have an appropriate timing device having a valid certificate of calibration.

d) **Standardizing indenters:** All calibrations of testing indenters shall be conducted with standardizing indenters. The calibration agency shall have one or more standardizing indenters appropriate for each Rockwell hardness scale listed under the calibration agency's scope of accreditation. Each standardizing indenter shall be obtained from NIST or have been calibrated by a calibration agency accredited under the requirements of this Guide.

e) **Verification of indenter balls:** When the scope of accreditation includes standardization of ball indenters, the calibration agency may either certify the chemistry, diameter, and hardness of the balls, or obtain a certificate or letter from the ball manufacturer stating compliance to the relevant tolerances for these measurements.

    1) Historically, Rockwell indenter balls have been obtained from companies that manufacture balls for many applications, such as for bearings. Generally, these manufacturers do not produce balls specifically for the Rockwell indenter application, and thus are not likely to seek NVLAP accreditation for measuring these characteristics. As a result, a manufacturer's certificate stating that the balls meet the requirements for chemistry, geometry and hardness is acceptable for these measurements, even if the ball manufacturer is not NVLAP-accredited to conduct the measurements.

    2) The destructive nature of a chemical analysis or a hardness test on individual indenter balls requires that verification testing be conducted on samples from a batch of balls. Thus, it is acceptable for indenter balls to be batch certified for chemistry, diameter, and hardness. A suitable sampling plan for certifying a batch of balls should be used to ensure that good process control was utilized in the production of the balls.

    3) For a calibration agency that performs these measurements itself, the calibration agency shall have instruments that are capable of measuring the diameter and the hardness of the ball as specified in

the corresponding test method standard. Each measuring instrument shall have a valid certificate of calibration.

f) **Standardized test blocks:** The calibration agency shall maintain an inventory of standardized test blocks at the Rockwell scales and hardness levels used for conducting standardizations of indenters.

g) **Monitoring test blocks:** The calibration agency shall maintain an inventory of monitoring test blocks in the required ranges for each Rockwell scale that the calibration agency conducts hardness measurements as part of the indenter standardization procedures.

h) **Verification of the geometrical and physical attributes of diamond indenters:** When the scope of accreditation includes the verification of the geometrical and physical attributes of diamond indenters, the calibration agency shall have suitable instruments for the measurement of the relevant parameters. The measuring instruments shall have the appropriate uncertainty, as specified in the relevant test method standards, and shall have a valid certificate of calibration. A calibration agency requesting accreditation for only the verification of the geometrical and physical attributes of diamond indenters is not required to have the instruments and reference materials listed in the preceding paragraphs with the exception of temperature and humidity measuring instruments.

2.11.6.5 Test and calibration methods and method validation

a) Indenters are standardized by verifying both the indenter's geometric and physical parameters and the indenter's performance on selected Rockwell scales. The calibration agency shall follow the standardization requirements and procedures specified in the relevant test standards. The calibration agency shall have documented procedures for conducting the standardizations of indenters, as specified in the relevant test standards.

b) **Performance verifications:** All indenters are verified for performance by conducting hardness tests on multiple Rockwell scales and hardness levels as specified in the relevant test standards.

1) **Standardizing indenters:** Standardizing indenters are generally verified for performance by comparison testing with a national primary standardizing indenter when a mechanism is available to make these comparisons. Such a comparison testing program is usually conducted by the national standardizing laboratory (NIST in the U.S.). When a mechanism is not available for comparisons with the national primary standardizing indenter, standardizing indenters may be verified by performance testing using primary standardized test blocks. When primary standardized test blocks are not available, test blocks may be verified by performance testing using standardized test blocks that were standardized by a calibration agency accredited by the procedures of this Guide.

2) **Testing indenters:** In all cases, testing indenters are standardized for performance by comparison testing with a standardizing indenter.

3) **Calibration offset values:** Standardization of indenters may include determination of hardness performance offset values with respect to the performance of the primary standardizing indenter or standardizing indenter that it was compared with. If such offset values are determined and reported as part of the calibration certificate, the calibration agency shall have documented procedures for determining these values.

2.11.6.6 Assuring the quality of test and calibration results

a) The calibration agency shall monitor the performance of the standardizing machine between indirect verifications. The monitoring procedures shall be in accordance with the appropriate test method standard when requirements are stated. The monitoring procedures should involve measuring monitoring test blocks each day that standardizations are made, and tracking the performance of the standardizing machine using control charts. Control charts are intended to indicate a loss of measurement control in the performance of the standardizing machine.

b) Each geometrical and physical measurement of the indenter must have an uncertainty small enough for the purpose.

c) Estimates of uncertainty of performance measurements should include all components that are evaluated by statistical methods (Type A), and all components that are evaluated by other methods (Type B). Sources of uncertainty in the standardization of indenters include, but may not be limited to:

   1) Standardization by comparison with a standardizing indenter:

      a. repeatability of standardizing machine,

      b. error in the standardizing indenter performance,

   2) Standardization by performance comparison with standardized test blocks (standardizing indenters only):

      a. all standardizing machine errors, and

      b. uncertainty in the standardized test blocks.

d) Statements of measurementuncertainty shall be consistent with NIST Technical Note 1297.

2.11.6.7 Handling of test and calibration items

a) Prior to calibration, the indenters shall be placed in the test environment, and allowed sufficient time to reach thermal equilibrium.

b) Prior to and after standardization, the indenters shall be stored in a manner that protects against mechanical or corrosive damage.

c) The standardized indenters should be packaged in a manner that adequately protects against mechanical or corrosive damage.

2.11.6.8 Records

Records must be kept for each indenter that is calibrated. These records must be in accordance with applicable test method standards, but should include the following:

a) indenter serial number,

b) indenter manufacturer,

c) date of manufacture and/or date of purchase,

d) date of standardization,

e) standardizing machine serial number,

f) standardizing indenter serial number,

g) personnel making measurements (indenter geometry and performance),

h) standardization test cycle (dwell times and force application times or velocities),

i) standardizing temperature,

j) applicable test method standard,

k) all standardization measurement values, and

l) offset values when determined.

2.11.6.9 Reporting the results

The certificate and standardization report shall be prepared by the person conducting the standardization and shall include all information as required by NIST Handbook 150 and the relevant test standard.